ECL USER'S MANUAL

Environmental Chemistry Laboratory
California Department of Toxic Substances Control
700 Heinz Ave, Berkeley CA 94710

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INTRODUCTION

The reliable measurement of environmental pollutants is essential in making decisions for waste management and public health protection. The measurement of these pollutants in multimedia is by no means a trivial task. Given that the physical and chemical properties of certain wastes are inherently complex, producing reliable data on the distribution and concentration of pollutants in the environment is an arduous task, often requiring specialized training. As a result, sampling and analysis can be the most expensive and time consuming aspects of a project. More importantly, errors in sample collection, sample handling, or analysis can invalidate projects or add to the overall project costs.

The ECL User's Manual was written to provide sampling guidelines as well as information on the efficient use of laboratory services. This document was written by staff of the Environmental Chemistry Laboratory (ECL) and the Public Health Institute (PHI) with input from other programs in the Department of Toxic Substances Control (DTSC), and the Environmental Chemistry Laboratory - Southern California (ECL-SC).

1.0 PLANNING

The successful collection of environmental data requires planning. The following planning guidance is based on the current quality system documents from the U.S. Environmental Protection Agency (EPA) (Ref 1).

1.1 QUALITY MANAGEMENT PLAN

A quality management plan (QMP) outlines or specifies all quality management activities for a specific program or organization (Ref 2). The EPA QMP requires conformance with ANSI/ASQC Standard E4 (Ref 3). The QMP defines the policies, criteria, roles and responsibilities for a program. The guidance in this User's Manual is consistent with the current US EPA-approved quality plan.

1.2 QUALITY ASSURANCE PROJECT PLAN AND DATA QUALITY OBJECTIVES (DQOs)

A quality assurance project plan (QAPP) is a project-specific document which includes project objectives plus sampling and analytical methods needed to accomplish the objectives. A critical part of the process is the development of **Data Quality Objectives** (DQOs), which are project-specific objectives, based on the acceptable levels of error for the project. Guidance for DQOs development is described in Reference 4.

DQOs must include both the process needed to obtain the product, and quality specifications of the product. DQOs should specify the quality of the data required to support the decisions making by the regulatory agencies. The DQO development process consists of the following seven steps:

Step 1. State the Problem
Step 2. Identify the Decision
Step 3. Identify the Inputs to the Decision
Step 4. Define the Study Boundaries
Step 5. Develop a Decision Rule
Step 6. Specify Acceptable Limits on Decision Errors
Step 7. Optimize the Design for Obtaining Data

In the DQO development process, the decision makers, data users, and individuals responsible for data collection should be identified and communication among the parties should be established. Continual communication among these parties is essential to the success of the project. Next, all available information on the site should be evaluated to gain an understanding of the site. With this information in hand, the current situation at the site should be described. Finally, the problems to be solved are identified.

In step 3, the tasks at hand as seen by the data users are stated and then the data types such as physical and chemical parameters are identified; the data quality needs are specified. These include prioritization of data uses, contaminants of concern, levels of concern (e.g., hazardous waste criteria, risk-based screening levels, etc.), required quantitation limits, and critical measurements. Next the sampling and analysis options are evaluated to maximize the use of the data. Finally, the precision, accuracy, representativeness, completeness, comparability, and other data quality indicators are specified.

The final step is optimizing the design of the data collection program. Here data collection components are assembled and needed measurements are listed.

Once DQOs are developed, a QAPP and/or SAP is prepared to document the activities to ensure that data collection programs will produce data of the type and quality required to satisfy the DQOs. The role of the regulatory agencies is to review, comment, and approve QAPPs and SAPs. The actual preparation of the plans are often the responsibility of contractors or responsible parties.

A project plan should address the items stated below. This manual provides guidance for the essential elements of a project plan. Regardless of the size of the project, a project plan should establish the objectives and scope of the project and define how these objectives will be accomplished.

The development and implementation of project plans may require a multi-disciplinary approach. Regulatory agencies must take the necessary precautions to assure that the overall integrity of a study is maintained during all its phases. A QAPP may be submitted as part of a Sampling and Analysis Plan (SAP) or as a separate document.

The current EPA QAPP guidance (Ref. 5) includes these elements:

A. Project Management
B. Measurement/Data Acquisition
C. Assessment/Oversight
D. Data Validation and Usability

The information required for an actual project will depend on the nature of the work.

1.3 SAMPLING AND ANALYSIS PLANS or (Field Sampling Plan)

Sampling and analysis plans (SAPs) are specific for each sampling and analysis episode.

Guidance for the preparation of these plans are at the local level but includes all monitoring and measurement activities specific for that episode.

The User's Manual is primarily a guidance to QAPPs and SAPs. The success of a project depends largely on planning done prior to initiating the project. QAPPs and SAPs serve as the cornerstone for the data collection activity and involve thought, research and coordination. Such plans allow the planned activities to be carried out in an organized manner in a pro-active mode rather than a reactive mode.

1.4 Waste Analysis Plans

Waste Analysis Plans (WAPs) are required for permitted facilities; A Guidance Manual can be found in reference 6.

1.5 REFERENCES

1) "Guidance for Developing Quality System for Environmental Programs,"EPA QA/6-1, EPA/240/R-02/008, November 2002 see: http://www.epa.gov/quality/qs-docs/g1-final.pdf

2) "Requirements For Quality Management Plans", EPA QA/R-2, EPA/240/B-01/002, US EPA Office of Environmental Information, EPA QA/6-1, EPA/240/B-01/002 Washington, DC 20460, March, 2001. See: http://www.epa.gov/quality/qs-docs/r2-final.pdf

3) American National Standard for Quality, "Specifications and Guidelines for Quality Systems for Environmental Data Collection and Environmental Technology Programs," ANSI/ASQC E4-1994, January 3, 1995.

4) US EPA, "Guidance for the Data Quality Objectives Process", EPA QA/G-4, August 2000, US EPA Office of Environmental Information, Washington, DC 20460, see http://www.epa.gov/quality/qs-docs/g4-final.pdf

5) US EPA, "EPA Requirements for Quality Assurance Project Plans" EPA QA/R-5, March 2001, see http://www.epa.gov/quality/qs-docs/r5-final.pdf

6) "Waste Analysis at Facilities that Generate, Treat, Store, and Dispose of Hazardous Waste, A Guidance Manual, OSWER 9938.4-03, April, 1994 http://www.epa.gov/compliance/resources/policies/civil/rcra/wasteanalygman-rpt.pdf

2.0. FIELD MEASUREMENT

Although environmental measurement has traditionally been done in the laboratory, data collection can often be optimized by including field measurement. Field measurement may include qualitative tests or quantitative tests.

2.1. FIELD TESTS

Field testing of concentrated industrial wastes has several advantages:

1) Site Safety Plans can be refined with better knowledge of the hazards of the wastes on site.

2) Sampling strategies can be more effective if some qualitative information on the waste is available. For example, if drums or other containers can be grouped into lots of the same material, then a stratified sampling strategy can be used. This approach is discussed further in Section 3, Sampling.

3) Incompatible wastes can be identified for future waste handling purposes.

4) Laboratory analytical requests can be more targeted and therefore more effective. More targeted analytical requests have led to shorter turnaround times and lower costs.

2.1.1. Safety Considerations.

In general, personal protection during field testing should be the same as that used for hazardous waste sampling. The use of a field laboratory with an adequate hood (with a face velocity of 60-100 linear feet per minute) can eliminate the need for most respiratory protection. However, gloves and goggles or face shield should always be worn when conducting tests. It is recommended that persons performing field sampling or testing should receive training on hazardous materials incident response operations.

2.1.2. Field Characterization Procedures.

Field characterization procedures range from a few screening tests, such as those listed below, to the elaborate HAZCAT (Hazard Categorization) scheme which was developed by Robert Turkington, formerly of CAL-OSHA. The sampling techniques for field testing are generally the same as for laboratory samples. For liquids, glass thieves are generally adequate for sample collection. Additional information on sampling is available in Section 3.3. Industrial Waste Sampling.

Explosivity Meter Test:

Follow the instrument manufacturer's directions for calibration and use. For waste in drums or other containers, insert the probe into a bung hole or other opening. A reading >100% of the Lower Explosive Limit (LEL) indicates that the contents are ignitable.

Organic Vapor Tests:

Portable direct-reading instruments such as an Organic Vapor Analyzer (OVA), or an HNU® with a photoionization detector (PID) and/or flame ionization detector (FID) can provide useful information on headspace or vapor samples. The instruments must be calibrated according to the manufacturer's directions. Positive results indicate that the waste contains volatile organic components. Record the readings and the calibration substance.

Physical Characteristics:

Note the physical state (liquid, solid, semisolid, or mixture). Note the color, viscosity (resistance to flow), and appearance (e.g., crystalline or amorphous). Note if any reaction with air is occurring, such as fuming, color change, or polymerization.

Radioactivity:

Use a radiation survey meter to check for an increase over background radioactivity.

Water Reactivity/Solubility:

Caution: This test should be performed in a hood with the sash as far down as possible or with adequate personal protection. Position test sample downwind from operator. Place about 1 gram (1 ml of a liquid) in a disposable glass or plastic container which contains 10 ml of distilled water and observe any reactions. Gas generation, color change, large temperature increase, ignition, or rapid polymerization are all signs of water reactivity. If a liquid sample mixes with the water to form a single phase liquid, it is considered to be miscible with water. If the liquid is not miscible with water, note whether it is denser (sinks) or lighter (floats). If a solid dissolves in water, it is considered to be water soluble. After it is determined that no reaction has occurred, measure the temperature with a thermometer or thermocouple (ref: ASTM D 5058-90 Test Method C).

Compatibility:

Caution: This test should be performed in a hood with the sash as far down as possible or with adequate personal protection. Position test sample downwind from operator. Combine samples of the two wastes in the ratio to be mixed, and observe any physical

changes. To determine potential exothermic reactions, measure the temperature before and after mixing. (Ref: ASTM D 5058-90 Test Method A).

pH:

For an aqueous (water-based) liquid, pH test strips can be immersed directly into a 5-10 ml sample in a disposable container. For non-aqueous liquids and solids, the sample must first be mixed with water or the pH test strip must be wetted first. Compare the color of the pH test strip with the color chart supplied with the strips. Due to the uncertainty of this test, a sample with a pH less than 3 should be considered acidic, and a pH greater than 10 should be considered alkaline (Ref: EPA Method 9041A).

Oxidizer Test:

Dip a strip of acidified potassium iodide (KI) paper into the sample or the sample mixed with water. A purple to black color is positive for a strong oxidizer.

Sulfide Test:

Dip a strip of acidified lead acetate test paper into the sample or the sample mixed with water. A brown or black color is positive for sulfides. Alternately, sulfides can be detected by acidifying a small sample with sulfuric acid and testing the headspace with a hydrogen sulfide detector tube, such as a Draeger® tube.

Cyanide Test:

Cyanides can be detected either by using a cyanide test paper (e.g., Cyantesmo test paper (Macherey-Nagel)) above an acidified sample, or by using a hydrogen cyanide detector tube (e.g., Draeger tube) above an acidified sample.

PCBs in Oil

The CLOR-N-OIL™ kit can be used to screen for halogenated organics in oil. It is a semiquantitative test; that is, it gives an estimate of the concentration of PCBs present. The test kit is available from the following:

Address:	Dexsil Corporation, One Hamden Park Drive, Hamden, CT 06517
Phone:	800 433-9745
FAX:	203 248-6523
Internet address:	http://www.dexsil.com

Sample Number: _____ Ambient Temperature: _____

Type of Container:_____

Markings on Container:_____

_____

Air Reactive: Pos._____Neg._____Radioactive: Pos._____Neg._____

Beilstein's Halogen Test: Pos._____Neg._____Liquid:_____

Solid:_____Other (Specify):_____

Appearance of Waste:_____

OVA or HNU Reading:_____(attach chromatogram if applicable)

_____

Waste/water mixture (determine the water reactivity, record liquids as soluble or insoluble in water, estimate the percent solubility of solids):

_____

_____

pH determination:_____

Oxidizer Test: Pos._____Neg._____ Peroxide Test:Pos._____Neg._____
Sulfide Test: Pos._____Neg._____ Cyanide Test: Pos._____Neg._____

Comments:_____

_____

_____

_____

Hazardous waste characterization data sheet.
Figure 2.1-1

Halogens in Oil

EPA and California have standards for total halogens (organic and inorganic halogens) in oil. The CLOR-D-TECT™ kit can be used to screen oil samples for halogens. The kit is available from:

Address:	Dexsil Corporation, One Hamden Park Drive, Hamden, CT 06517
Phone:	800 433-9745
FAX:	203 248-6523
Internet address:	http://www.dexsil.com

The Beilstein test can also be used to estimate organic halogens.

Lead Test Kits

Several lead test kits are available for the testing of surfaces, soil, and ceramic ware. One test kit is the "Lead Check" swab, available from:

Address:	HybriVet Systems, Inc., Framington, MA 01701
Phone:	1-800-262-LEAD
Internet Address:	http://www.leadcheck.com

When evaluated using soil spiked with lead-based paint, the Lead Check test kit results correlated well with the hazardous waste classification using the California Waste Extraction Test.

2.1.3. Reliability of Field Tests.

Field tests are normally designed to err on the positive side; that is, to produce a higher rate of false positives than false negatives. However, an excessively high false positive rate can lead to high analytical costs for laboratory verification and overly expensive waste handling plans. New field tests should be evaluated against the established laboratory methods prior to field use.

2.1.4. Scope of Field Tests.

Field tests are generally more successful on relatively pure substances, because appearance may give some clues and because the field tests give more definitive results on pure substances. Conversely, complex waste mixtures may not yield useful information from field tests.

2.1.5. Commercially Available Test Kits.

A number of field test kits are available for water and wastewater, such as from Hach Kits:

Address:	Hach Chemical Company, P.O Box 389, Loveland, CO 80539-0389
Phone:	303 669-3050
Internet Address:	http://www.hach.com

Color indicator tubes (e.g., Draeger® tubes) are available for a variety of airborne contaminants. Follow the manufacturer's instructions and note potential interferences when interpreting the result. Newer test kits based on immunoassays, are available for some pesticides and industrial chemicals. Consult the manufacturer's information for the kit's applicability and limitations.

2.2 REFERENCES

1) "Standard Test Methods for Compatibility of Screening Analysis of Waste," ASTM D 5058 - 90, in Annual Book of ASTM Standards, American Society for Testing and Materials, July, 1990.

2) "Field Analytical Measurement Technologies, Applications, and Selection", California Military Environmental Coordination Committee (CMECC), April 1996.

3.0 SAMPLING

This section presents guidelines for the collection, preservation, and transportation of waste samples and environmental samples. This field is still dynamic, so guidelines will change as more research is completed on the behavior of contaminants in samples. This section was compiled from a number of sources; the primary sources are listed at the end of each subsection.

Questions regarding any of the topics covered here should be directed to the contacts listed in Section 4.0, or the appropriate reference should be consulted.

3.1 SAMPLING PLANS

Sampling is generally the source of most errors in environmental measurement. Controlling sampling errors to acceptable levels requires attention to the steps described below. Whenever feasible, the sampling and analysis procedures should be defined in a sampling and analysis plan (SAP), Field Sampling Plan (FSP), or other plan. Such plans should be written after DQOs have been developed (Section 1).

3.1.1 What is the Problem?

Sampling will likely not be successful unless the project objectives have been determined in advance. Two common objectives for sampling are waste classification and site assessment. Waste classification typically involves sampling waste streams (e.g., drums and piles), while site assessment involves sampling air, water, soil, soil gas, or other media which may have been contaminated by hazardous materials.

3.1.1.1 Waste Classification.

Waste classification typically answers the question: Is this waste a hazardous waste? In such cases, samples should be representative of the waste stream. That is, the average properties of the samples generally should provide an acceptable estimate of the average properties of the entire waste stream. The definition of representative sample from Title 22 is one that has the average properties of the waste being sampled. While this is sometimes a worthwhile goal, it is rarely possible to obtain one real world sample of an industrial waste which has the average properties of the entire waste.

Waste sampling and analysis may also be done to answer the question: What is it? This is often the case for sampling inadequately labeled drums, carboys, or other containers. Because information obtained in the field (e.g. field analysis) may tentatively identify substances of concern, sampling plans may need to be modified as information becomes available. For example, if field screening indicates that a group of drums contains the same waste type, a stratified random sampling may be appropriate, as will be discussed below.

Waste generators are directed by Title 22 to comply with Chapter 9 of SW-846 for sampling and sample handling. EPA has drafted a stand-alone sampling guidance document entitled "RCRA Waste Sampling Draft Technical Guidance." (http://www.epa.gov/epaoswer/hazwaste/test/samp_guid.htm) This guidance was proposed as a replacement for the current sampling guidance version of Chapter Nine found in EPA publication SW-846. EPA is currently reviewing comments on the document and will release the document after it is revised. Until the completion of the Technical Guidance, Chapter Nine will remain the applicable guidance.

The American Society for Testing and Materials (ASTM) has published numerous standards and guides for sampling. The ASTM documents are available at engineering libraries and from ASTM.

Mailing address: ASTM
1916 Race Street
Philadelphia, PA 19103

Internet address: http://www.astm.org/

3.1.1.2 Site Assessment.

Sampling for site assessment may be quite different than sampling for waste characterization. The questions of interest may be: "What is the type and extent of contamination?" or "Has disposal of a hazardous waste occurred?" The Data Quality Objective process can produce an optimum sampling and analysis plan based on available resources. For more information, refer to EPA's "Methods for Evaluating the Attainment of Cleanup Standards. Volume 1: Soils and Solid Media," EPA 230/02-89-042.

3.1.1.3. "Clean Closure" Soil Sampling.

A particular problem in sampling is how to determine whether a site has been cleaned up to either a cleanup level or to a "background" level. The purpose in this case is generally to determine whether contamination on site is greater than the cleanup or background level. In order to prepare an adequate sampling plan, it is necessary to establish in advance what cleanup criteria will be used. If "background" is to be used, either data can be collected from control areas near the site or existing ambient levels can be used, e.g., USGS Geological Survey data on elemental levels in uncontaminated land. The control areas should be selected which are similar to the site in soil type and proximity to other sources of contamination, such as freeways. Caution must be used in using published data from the U.S. Geological Survey and the University of California, as these data can be too sparse to apply to specific sites. Further these data are of total soil element concentrations and not comparable to "TTLC" concentrations which are not total but partial acid digestions.

For addition information in determining whether site soil contamination is significantly greater than "background", refer to EPA's "Statistical Methods for Evaluating the Attainment of Cleanup Standards. Volume 3: Reference-Based Standards for Soils and Solid Media," EPA 230-R-94-004. This guidance presumes that site soils can be compared to soils of a similar type that are uncontaminated.

For additional information on cleaning up a site to predetermined "cleanup levels", refer to

EPA's "Methods for Evaluating the Attainment of Cleanup Standards. Volume 1: Soils and Solid Media," EPA 230/02-89-042. (http://www.clu-in.org/download/stats/vol1soils.pdf)

The sections below discuss the problems of determining the number of samples and evaluating the results.

3.1.1.4 Groundwater monitoring.

Groundwater monitoring requires careful specification of sampling and analytical methods. The purpose of such monitoring should be clearly established in a written plan well in advance of the sampling. The choice of sampling techniques, filtration techniques, analytical methods, and quality control samples will depend on the purpose of the sampling, as discussed in Section 3.2. For guidance on groundwater sampling and data interpretation, refer to "Methods for Evaluating the Attainment of Cleanup Standards. Volume 2: Ground Water," EPA 230-R-92-014.
(http://www.clu-in.org/download/stats/vol2gw.pdf)

3.1.1.5 Air Monitoring.

Typical purposes of air monitoring are: (1) to determine the magnitude of a source by comparing upwind and downwind concentrations, or (2) to determine the average concentration of some substance over a specified area and time interval.

3.1.2 Standard Operation Procedures (SOPs) for Sampling.

Examples of sampling SOPs can be found at website
http://www.epa.gov/region4/sesd/eabsop/eabsop.pdf.

3.1.3 Sampling Strategies.

Once the purpose of sampling has been established, the appropriate sampling strategy can be chosen. As mentioned above, references are available for choosing sampling strategies, depending on the purpose. This section will briefly describe the most common strategies, along with the implications for laboratory analysis.

3.1.3.1 Authoritative Sampling.

In authoritative (also called targeted or judgmental) sampling, the person collecting the sample decides on the sampling locations- generally to find the area of highest contamination. In a number of situations, this is the strategy of choice to determine whether disposal has occurred or to determine whether more extensive sampling is warranted. This strategy requires the fewest samples and provides the smallest number of measurements

below detection limits. This is usually the technique of choice to find "hot spots" or to find evidence of past disposal. This type of design can be very effective if the collector is familiar and knowledgeable about the site, and if goal of sampling is merely to establish that contamination may exceed some set criteria.

3.1.3.2 Random Sampling.

Random sampling allows statistical inferences to be made about an entire waste or other area. For example, one can calculate the mean and standard deviation for the concentration of a substance, plus a confidence interval which contains the true mean concentration, at a given level of confidence. Random sampling may be simple, stratified, or systematic. All three strategies for random sampling typically involve placing a grid over a map of the area to be sampled.

1) Simple Random Sampling

Using random sampling, the sampling and analysis of all samples should be identical so that bias is minimized. Simple random sampling can involve placing one grid over a map of the entire area and randomly choosing grid cells to sample. This method results in estimates of parameters (e.g., mean and standard deviation) for the entire area.

2) Stratified Random Sampling

The sampling and analytical procedures for stratified sampling are generally the same as for simple random sampling, although the evaluation of the data is different. In this strategy, the soil or solid waste (i.e., the "population") is divided into subregions (i.e., "strata") that are believed to be internally more homogeneous. Each subregion is randomly sampled. Means and variances for each subregion are calculated and used to calculate an overall mean and variance for the population. The statistical methods used to estimate the overall mean and variance presume that data are normally distributed. Examples include differentiating between highly contaminated (hot spots) and less contaminated areas or between surface, intermediate, and bottom layers of an impoundment.

3) Systematic Sampling

Systematic sampling involves the collection of samples at predetermined, regular, intervals; for example, samples taken at ten foot intervals along a line from an outfall. This technique is often used, but can result in biased results if there are periodic variations in the material to be sampled. Systematic sampling also minimizes the effect of spatially correlated data on estimate of sample variance.

3.1.3.3 Composite Sampling.

Compositing of samples is an effective way to reduce analysis costs. From a statistical point of view, compositing of samples loses information which would have been provided by the individual samples, but reduces the variability per composite sample. Compositing also, in effect, raises the analytical detection limit for the individual sub-samples because each sample is diluted by the other samples in the composite. The effective detection limit of each subsample is:

$$DL_S = DL_C \text{ X } n,$$

Where DL_S = Detection limit for each subsample,
DL_C = Detection limit for the composite sample,
n = Number of subsamples in the composite sample.

For example, if the detection limit for lead in a composite soil sample was 1 mg/kg (ppm) and each composite was made from 5 subsamples, then the effective detection limit for lead in each subsample is (1 mg/kg)(5) = 5 mg/kg (ppm).

Composite samples are sometimes preferred over individual samples in order to reduce analytical costs by reducing the number of samples. If the detection of hot spots is the sampling objective, then composite sampling can yield significant savings if the probability of exceeding an action level is less than 10%. One approach which has been successfully used is to prepare composites from portions of the original samples and archive the remainder of the original samples. The results of the composite samples can then be used to determine what analysis, if any, is needed on the archived samples. The holding times for the analytical methods should be considered with this strategy.

For additional information refer to ASTM standard D 6051-96 titled "Standard Guide for Composite Sampling and Field Subsampling for Environmental Waste Management Activities". This standard may be obtained from ASTM through their web site: http://www.astm.org.

3.1.3.4 Homogenizing and Splitting Samples.

Splitting samples is often necessary to provide samples to a defendant or responsible party, to send blind duplicate samples to the same lab, or to send split samples to a reference lab. Homogeneous material, such as water or fine grained soil, can be easily split in the field. Moderately heterogeneous material, such as coarse-grained soils, can generally be homogenized and split in the field using an additional container. The materials used for homogenization should be the same as the materials recommended for sample

containers in Section 3.6. The homogenization of soil samples prior to volatile organics analysis can result in significant losses, so co-located samples should be collected instead of split samples. Extremely heterogeneous material, such as mixtures of oil and water, may need to be split in a laboratory. If necessary, contact the lab which will be doing the compositing to obtain their standard procedure for sample splitting. Chain of custody must be maintained on the material from the initial point of collection, through sample splitting, to analysis. Chain of custody will be discussed further in Section 3.9. For additional information refer to ASTM standard D 6323 98 titled "Standard Guide for Laboratory Subsampling of Media Related to Waste Management Activities". This standard may be obtained from ASTM through their web site: http://www.astm.org.

3.1.4 Statistical Considerations.

In some sampling situations, statistical parameters must be considered before sampling. These include estimates of the mean (average) and variance, calculation of the appropriate number of samples, and selection of appropriate sampling locations. Detailed procedures for data analysis can be found in *Guidance for Data Assessment,* EPA QA/G-9.
Internet address: http://www.epa.gov/quality/qa_docs.html

Both the required number of samples to be collected and the final statistical analysis depend on the purpose of the sampling. In Case 1, below, which is relevant to waste classification, the average concentration of the results for randomly collected samples, is compared to a regulatory threshold, e.g., a Total Threshold Limit Concentration (TTLC) or Soluble Threshold Limit Concentration (STLC). The object of this sampling is to determine whether the average waste concentration is above the set threshold. Case 2, which describes Site Assessment, involves both random sampling, as described in Case 1, and authoritative sampling, which is not usually amenable to statistical analysis. In Case 3, which is relevant to Clean Closure as well as other activities, the average sample concentration is compared to the average concentration of background samples. In this case, there is statistical uncertainty or variability in both the sample and background concentration means.

In either statistical treatment, the tradeoffs in deciding the number of samples are 1) the amount of data and the corresponding confidence in results, versus 2) the costs and time for sampling and analysis. Chapters 9 and 10 of SW-846 and other references (e.g., EPA's Sampling Quality Assurance User's Guide or ASTM D4687 Standard Guide for General Planning of Waste Sampling) provide guidance on selecting the appropriate minimum number of samples when the cost of sampling and analysis is considered. The following approaches do not explicitly consider cost, but the chosen level of confidence will influence cost.

Example 1 - Waste Classification

Here are examples of DQOs for a generator's waste classification.

Step 1: State the Problem. To properly manage waste, the generator must classify a wastestream according to California hazardous waste regulations.

Step 2: Identify the Decision. If a waste exceeds any of the Hazardous waste criteria, it will be classified as a hazardous waste.

Step 3: Identify the Inputs to the Decision. The waste in question, a burn dump ash, is known to contain elements, especially copper and lead, which could render it hazardous. Data are needed for total metals, Waste Extraction Test (WET)-metals, Toxicity Characteristic Leaching Procedure (TCLP)-metals on an adequate number of samples.

Step 4. Define the Study Boundaries. The vertical and horizontal extent of the burn ash is determined from available data, and field observations.

Step 5. Develop a Decision Rule. If the mean total concentration exceeds a Total Threshold Limit Concentration (TTLC); or if the mean extractable concentration exceeds the respective Soluble Threshold Limit Concentration (STLC) or TCLP Limit, the waste will be classified hazardous.

Step 6. Specify Acceptable Limits on Decision Errors. Based on the guidance in the SW-846 Field Manual, an 80% confidence interval will be used for waste classification. If the mean concentration exceeds the lower 80% confidence interval of the mean, the waste will be judged hazardous. There is a 10% chance that the true mean is greater than the upper 80% confidence interval, i.e., there is a 10% chance of a false negative decision.

To compare the mean concentration with a regulatory limit, the Student's t-test is often used. This treatment assumes that the samples are independent of each other and the contaminant concentrations have a normal distribution. In environmental samples, these assumptions may not be met. As Student's t-test is not very sensitive to small deviations from normal distribution, often the t-test may still be used.

If, however, there is a substantial deviation from normality, the data may have to be transformed to approximate a normal distribution. The most common transformation with environmental data is a logarithmic transformation (usually the natural logarithm, ln). The references should be consulted for details on statistical tests with log-transformed data.

In order to determine whether the average contaminant concentration is above the regulatory threshold with a certain level of confidence, the required number of samples is given by equation 3-1.

$$n = \frac{t_{1-\alpha}^2 s^2}{\left(RT - \bar{x}\right)^2}$$ *Equation 3-1*

In which t = Student's t statistic at the desired confidence level, 1-α, s = the sample standard deviation, RT = the Regulatory Threshold, and $\bar{x}$ = the sample mean. For California hazardous waste criteria, the RT may be a Total Threshold Limit Concentration (TTLC), a Soluble Limit Threshold Limit Concentration (STLC), or an established background level.

SW-846 specifies that $t_{0.2}$ should be read from a two-tailed t table, as presented in Table 3.1-1. This is numerically equal to $t_{0.1}$ read from an one-tailed t table.

Table 3.1-1 Tabulated Values of Student's "t" for evaluating Solid Wastes

Degrees of freedom (n-1)[a]	Tabulated "T" value[b]
1	3.078
2	1.886
3	1.638
4	1.533
5	1.476
6	1.440
7	1.415
8	1.397
9	1.393
10	1.372
11	1.363
12	1.356
13	1.350
14	1.345
15	1.341
16	1.337
17	1.333
18	1.330
19	1.328
20	1.325
21	1.323
22	1.321
23	1.319
24	1.318
25	1.316
26	1.315
27	1.314
28	1.313
29	1.311
30	1.310
40	1.303
60	1.296
120	1.289

[a] Degree of Freedom (df) are equal to the number of samples (n) collected from a solid waste less one.
[b] Tabulated "t" values are for a two-tailed confidence interval and a probability of 0.20 (the same values are applicable to a one-tailed confidence interval and a probability of 0.10).

Since this approach requires some previous knowledge about the distribution of contaminants, it can best be carried out when preliminary sampling has been performed and the results used to calculate the number of samples for the second sampling. The mean and standard deviation are estimated from the preliminary sampling. The appropriate t statistic is found in the table and the resulting number of samples required to achieve that level of confidence is calculated. The value for t in the row corresponding to (n-1) degrees of freedom is chosen. Because the value of t depends upon the number of samples collected, an iterative approach can be used in which a number of samples is postulated, the appropriate t chosen, and the number of samples is calculated according to Equation 3-1. This is repeated until the calculated n is as close as possible to the postulated n which corresponds to the t value. The number of samples is always rounded up to the larger number when a fractional number results. Four samples are considered to be the minimum number for waste classification advised by EPA in SW-846.

Step 7. Optimize the Design for Obtaining Data. The number of needed samples can be calculated from equation 3-1. However, since the objective is to estimate the mean concentration, composite sampling (3.1.2.3) can be used to reduce the number of samples required for analysis. When the final sampling is conducted, a few additional samples should be collected because if the standard deviation of the samples is higher than the standard deviation of the preliminary samples, the required n will be higher. These additional samples may be archived and analyzed only as needed, if the holding times permit.

If the average contaminant concentration is above the regulatory threshold, the waste is to be considered hazardous. If the average concentration is below the regulatory threshold, the waste may still be considered hazardous if the regulatory threshold falls within the confidence interval of the average concentration. That confidence interval is calculated according to Equation 3-2:

$$Cl = \bar{x} \pm t_{0.2} s / \sqrt{n} \qquad \textit{Equation 3-2}$$

In which $t_{0.2}$ is obtained from the two-tailed t-table presented in Table 3.1-1, $\bar{x}$ is the mean of the sample concentrations, s is the standard deviation of the sample concentrations, and n is the number of samples.

Use of the Student's t-test assumes that the contaminants are normally distributed across the site. If this is not the case (determined through use of a goodness-of-fit test), the data can be transformed by scaling all of the data values. The most common transformations are obtained by taking the logarithm or square root of each concentration datum. The resulting transformed data may have a normal distribution even if the original data did not.

An alternative to the two sample t-test is a non-parametric test; EPA G-9 or another reference on nonparametric statistics should be consulted.

3.1.5 Elements of the Sampling Plan.

Before a sampling project is begun, a sampling plan should be drawn up. Elements which should be included in such a plan are:

I. Objective and scope of the sampling
   - A. Brief description of site
   - B. Background and objective of monitoring
   - C. Personnel in charge of sampling

II. Sampling Overview
   - A. Map of site with sampling locations
     - sampling strategies
     - types of samples to be collected
   - B. Analyses to be performed (table)
     - aliquots (volumes/weights)
     - containers
     - preservatives
     - special handling
     - analytical methods to be used
   - C. Monitoring well details (if wells included)
     - well depth
     - screened interval
     - casing diameter
     - previous depth to water measurement
     - dedicated pumping equipment
     - estimated purge and recovery time
   - D. Sampling and shipping schedule

III. Presampling Procedures
   - A. Safety survey
     - instruments and procedures
     - concentration limits for each level of protection
     - clothing and other protective equipment
   - B. Well preparation (if wells included)
     - physical measurements (depth to water, etc.)
     - well purging (volume, method, disposal)
     - procedure for slowly recharging wells
   - C. Field measurements (parameters, methods)

IV. Sample Collection
   - A. Equipment and procedures
   - B. Order of collection

V. QC Samples (types, numbers, procedures, locations)

VI. Decontamination
- A. Equipment (procedures, schedule)
- B. Personnel decontamination procedures

VII. Documentation
- A. Logbooks
  - required inclusions
  - assigned person
- B. Photographs

VIII. Chain of Custody requirements

IX. Labeling and packaging of samples

X. Transportation

Several of these elements can be used with only minor modifications for many different projects. Other elements may need to be written specifically for each project.

3.1.6 References.

1) U.S. EPA, "Test Methods for Evaluating Solid Waste - Physical/Chemical Methods," SW-846, 3d ed. SW-846 is available online at: http://www.epa.gov/epaoswer/hazwaste/test/main.htm

2) Gibbons, J. D., Nonparametric Methods for Quantitative Analysis (Second Edition), American Sciences Press, Columbus, Ohio, 1985.

3) Gilbert, Richard O., Statistical Methods for Environmental Pollution Monitoring, Van Nostrand Reinhold Co., New York, 1987.

4) Keith, Lawrence K., "Principles of Environmental Sampling," Environ Sci Technol, Vol 24, pp 610-617, May, 1990.

5) ASTM: D4687, Standard Guide for General Planning of Waste Sampling.

6) RCRA Waste Sampling Draft Technical Guidance, 2002 http://www.epa.gov/SW-846/samp_guid.htm

3.2 WATER SAMPLING.

3.2.1 Groundwater.

The following section briefly describes sample collection from groundwater monitoring wells and is not comprehensive. For further information, see Appendix B of Guidance Document, Monitoring Requirements for Permitted Hazardous Waste Facilities (GSU, 2001), Representative Sampling of Ground Water for Hazardous Substances (CALEPA, 1995), Practical Handbook of Ground-Water Monitoring (Nielsen, 1991), and Low-Flow (Minimal Drawdown) Ground-Water Sampling Procedures (Puls and Barcelona, 1996) the source for much of this section.

3.2.1.1. Detection of Organic Vapors in Well Headspace.

It may be necessary to monitor the air at and around the well head immediately upon opening the well using a direct reading instrument such as an organic vapor analyzer (OVA), photoionization detector (PID), and/or a combustible gas indicator (CGI) to determine the potential for fire, explosion, or other health and safety hazards. Detailed plans and acceptable limits should be included in the health and safety portion of the sampling plan.

3.2.1.2 Measurement of Static Water Level Elevation.

Routine measurement of static water level elevations are important to determine whether the predicted horizontal and vertical flow gradients have changed since last measured. Measurements should include the depth to standing water and often the depth to the bottom of the well casing. This information may be needed to calculate the volume of water to purge from a well and to provide a check on the integrity of the well (e.g., to identify problems with siltation). Measurements should be made on all wells the first day at the site prior to pumping any well, and again at a well prior to sampling it. On certain sites, water level measurements should be made within a relatively short period of time (i.e., tidally influenced aquifers, aquifers with very flat gradients, etc. [see GSU, 2001]). Instruments used to make the measurements should be capable of obtaining accurate readings to within ± 0.01 foot. An electronic depth-sounding device is preferred for measuring the depth to water. Measurements should be referenced to a marked point, usually the top of the well casing, whose elevation has been surveyed by a licensed surveyor. The depth measuring device must be thoroughly cleaned between wells to prevent cross contamination and maintain sample representativeness (see Section 3.2.4).

3.2.1.3 Detection of Immiscible Layers.

If present in high concentrations, relatively insoluble organic liquids may form either a floating phase (light non-aqueous phase liquid [LNAPL]) on top of well water or a sinking dense layer (dense non-aqueous phase liquid [DNAPL]) depending on the density of the liquid. Samples collected from within the well may contain a mixture of both of these layers and consequently be representative of neither the contaminant layer nor the bulk of the well

water. Determination of the presence of these layers is important in interpretation of ground water data as well as in evaluation of subsurface transport and mitigation measures. Organic liquid-water interface probes can determine the existence and thickness of these layers by lowering the probe into the well before purging or sampling. As a supplement to the interface probe, a transparent bottom-opening bailer can also be used to detect and collect floating layers. If an immiscible layer is detected, its thickness should be recorded and a sample collected.

3.2.1.4 Well Evacuation.

There are essentially two accepted methods for purging groundwater from high and moderate yielding wells: purging large (i.e., three well volumes) or small (i.e., low-flow micropurging) volumes of water from a well prior to sampling.

The concept behind large volume purging is to remove standing water from a well that may not be representative of in-situ ground water quality. Removal of standing water from a well allows fresh ground water from the formation to replace it. The standing water should be drawn down from near the water surface to ensure that fresh water entering the well screen will move upward. While it is generally accepted that well water in the well casing is not representative of the formation water, water in the screened interval of the well may in fact be representative. Low-flow purging/sampling is conducted based on this assumption, but is dependent upon well construction and site hydrogeology. Low flow sampling is a technique that minimizes the hydraulic stress on the aquifer during purging and sampling. This is done by pumping from the screened zone at a low flow rate that will cause minimal drawdown of the water level in the well. Drawdown is measured in the well concurrent with pumping using a water level meter. The use of bailers for purging or sampling is not acceptable for low-flow purging/sampling and is generally discouraged for all types of groundwater sampling.

Large and small volume purging do not require a specific flow rate or purge volume as they are dependent on aquifer conditions. A sample can be collected after the water level and measured field parameters (pH, specific conductance, dissolved oxygen, oxidation-reduction potential, temperature, and turbidity) stabilize over three consecutive readings taken at appropriate intervals. The actual volume of water purged should be based on stabilization of field parameters.

Sampling low yielding wells is problematic. Low yielding wells are defined here as wells that can not sustain a static water level during groundwater extraction at a rate of 100 milliliters per minute. It will be possible to completely drain the well using a low pumping rate if the pump draws from the bottom of the well. Exposing the well intake may cause volatilization or chemical reactions to occur resulting in non-representative samples. At no time should the pumping rate be so great as to cause ground water to cascade down the intake screen into the well for either low flow or conventional sampling. Comparative side-by-side sampling is strongly recommended for low yielding wells. For example, in the case of volatile organic compound (VOC) sampling, comparisons could be made between no

purge (i.e., diffusion bags, no purge micropurge), low flow techniques, and large well volume purging. Samples to be analyzed for volatile components should be collected as soon as sufficient water has reentered the well.

Whenever possible, purge rates should not exceed aquifer recharge rates determined from appropriate well testing. Wells should be purged at rates below those used to develop the well to prevent agitation of sediment, to prevent damage to the well, and to avoid disturbing accumulated corrosion or reaction products in the well. A low flow rate will reduce the possibility of volatilizing organic compounds from the water and reduce the likelihood of mobilizing solids in the subsurface that are immobile under natural flow conditions.

Large volume purging collects groundwater from a larger radius around a well screen than does low volume purging. For this reason one should consider if low flow sampling should be compared to large volume purging. If contaminants are only detected by the large volume method, this may suggest the well is not appropriately located for low flow sampling.

Purging of wells is routinely done with submersible positive displacement pumps (i.e., bladder pumps, centrifugal pumps). Use of dedicated equipment is preferred to minimize risk of contaminant introduction into the well and samples, minimize well disturbance and sampling artifacts, reduce the need for sample filtration, minimize the time spent sampling, and reduce the number of equipment blanks. Refer to CALEPA (1995) and Nielsen (1991) for a detailed discussion of different kinds of groundwater sampling devices.

Provision must be made to dispose of the purged water properly.

3.2.1.5 In-Situ or Field Analyses.

Several ground water parameters are physically or chemically unstable and should be tested either in the well, in the sampling/purging line using in-line probes, or immediately after collection using field meters. Examples of unstable field parameters include pH, dissolved oxygen, oxidation-reduction potential, temperature, and turbidity. The preferred method of making these measurements is to insert probes directly into the purge line so that a continuous reading can be taken as water is purged from the well. The final reading is taken when stable values are attained. If in-line probes are not available, then another alternative method is a "down-hole" or in-situ probe which measures field parameters in the well water. Otherwise, groundwater should be brought to the surface during purging, collected in a sample container or cup, and measured quickly with as little contact with the atmosphere as possible.

Field notes and logs taken at this time should include the appearance of the purge water including its color, turbidity (measured in nephelometric turbidity units - NTUs), and odor. Samples which are turbid may not be suitable for analysis and may be indicative of well damage, improper well design, or need for well development.

3.2.1.6 Sample Withdrawal.

Sampling equipment must be constructed of inert material and be used to minimize sample disturbance resulting in changes in water chemistry. Wells should be sampled from least to most contaminated.

There are four broad categories of groundwater sampling devices: grab samplers (e.g., bailers and syringe devices), positive displacement pumps (e.g., bladder and centrifugal pumps), suction lift pumps (e.g., direct line and peristaltic pumps), and gas contact pumps (e.g., gas-lift and gas-drive devices). The CALEPA (1995) document summarizes the capabilities of these different groundwater sampling devises. Table 1 of the 1995 CALEPA document indicates that grab, suction lift, and gas contact devices can be unsuitable for obtaining representative groundwater samples, especially for volatile constituents. To encourage innovation, CALEPA may allow the use of other sampling methods provided it is demonstrated that the method can yield representative groundwater samples on a site-specific basis.

Care should be taken at all times not to agitate ground water in the well or samples allowing volatile or gaseous material to escape. Sampling equipment (especially bailers) should never be dropped into the well or allowed to leak water back into the well as this can cause degassing of the water upon impact.

Ideally, enough purging, sampling, and filtering equipment can be taken to the field so that each item is used in only one well and taken to the lab for thorough decontamination (see Section 3.2.4). If a piece of equipment must be used in more than one well, it must undergo field decontamination procedures and equipment blanks should be collected (see Section 5.0). Clean, powderless gloves should be worn by sampling personnel and should be changed often. A clean plastic sheet should be placed around the well to prevent surface soils from coming in contact with purging equipment and lines, which in turn could introduce contaminants to the well. A plastic bag may be pulled down over the top of the well casing and samples collected through a hole in the bag for further protection during sampling.

Samples should be collected and containerized in the order of volatilization. A collection order recommended by CALEPA (1995) is:

- VOCs
- Semivolatile organic compounds
- Major water quality cations and anions
- Stable isotopes (e.g., oxygen, hydrogen, nitrogen, lead)
- Metals
- Cyanide
- Turbidity
- Radionuclides

3.2.1.7 Filtration.

If turbidity is less than 5 NTU, filtering is not necessary. Samples should never be filtered when a water supply well is sampled. For risk assessment purposes and to assess facility impacts to groundwater, unfiltered samples should also be considered if significant colloidal transport is suspected (Filtered samples may also be collected at the same time for comparison). Filtered samples for dissolved metals analysis should be used whenever groundwater is excessively turbid or, in some cases, to reduce statistical outliers. Poorly designed or developed wells yielding inappropriately high turbidity values should be replaced if re-development does not improve turbidity conditions. Filtration should also not be used to compensate for poor sampling practices.

When filtering is conducted, use of in-line filters is strongly recommended since filtering after groundwater contacts the atmosphere can underestimate metal concentrations due to possible precipitation of metal oxides. The use of 1 micron filters is recommended to allow passage of colloids and filtration of particles greater than clay size. Filters must be discarded after use at each well.

In those instances where in-line filtration is not possible, it may be advisable to collect both filtered and unfiltered samples. Filtering should be done as quickly as possible using positive pressure filtering equipment (laboratory filtration or filtration by vacuum methods are not acceptable).

It is essential that when a sample is filtered for metals, no preservatives/acids be added until after filtration as this will tend to dissolve particulates. Filters should be pre-washed per manufacture's instructions.

3.2.1.8 Field Logs.

One member of the sampling team should be assigned to make observations and record information in field logs and notebook.
Observations recorded should include:

- Collectors' names
- Well identification
- Static water level depth and measurement technique
- Presence of immiscible layers, detection method, and thickness
- Well yield, high or low
- Evacuation procedure/equipment
- Purge volume, pumping rate, and time purged
- Appearance of ground water (color, turbidity, odor, etc.)
- Sample collection procedures/equipment
- Date and time of collection
- Sample treatment and containers
- Parameters requested for analysis
- Field analysis procedures and results

- Sample packing, distribution, and transporter
- Other field observations including climatic conditions and problems encountered
- Photographs
- Date and title of approved sampling plan

3.2.2 Seeps and Springs.

Seeps and springs are generally areas where the ground surface intersects the water table. Because of reactions of the water with microbiological populations and the atmosphere, oxygen content, pH, nutrient and metal concentrations may be quite different from those in the ground water. However, seep and spring analyses can be used in risk assessments and as evidence of contaminant migration if properly interpreted. A scoop, or dipper/pond sampler can be used to collect samples from seeps. The sampler can be gently suspended in the water or laid against the bank and the water will flow with very little additional disturbance into the sampler for transfer to sample bottles. It is important to collect the sample as close to the actual seep as possible to reduce contact time with the atmosphere and potential for surface contamination. In some cases, soil may need to be removed, with appropriate decontaminated tools, up to a foot or two to get enough flow to collect in the sampler. At sites where seeps are repeatedly sampled, it may be advantageous to install shallow wells (<5 feet) which are then purged and sampled as regular monitoring wells.

3.2.3 Surface Waters.

Whereas in ground water sampling the sampling points are determined upon well construction, both location and depth of samples from surface waters must be decided upon before sampling. Usually, standing water (e.g., ponds) cannot be assumed to be well-mixed or uniform in composition. Therefore, the type of sampler used and the care with which it is placed becomes more important. References which discuss the choice of sampler include Ford et al., 1984 (from which much in the following paragraphs is drawn), the U.S. Department of the Interior, 1977, and deVera et al., 1980.

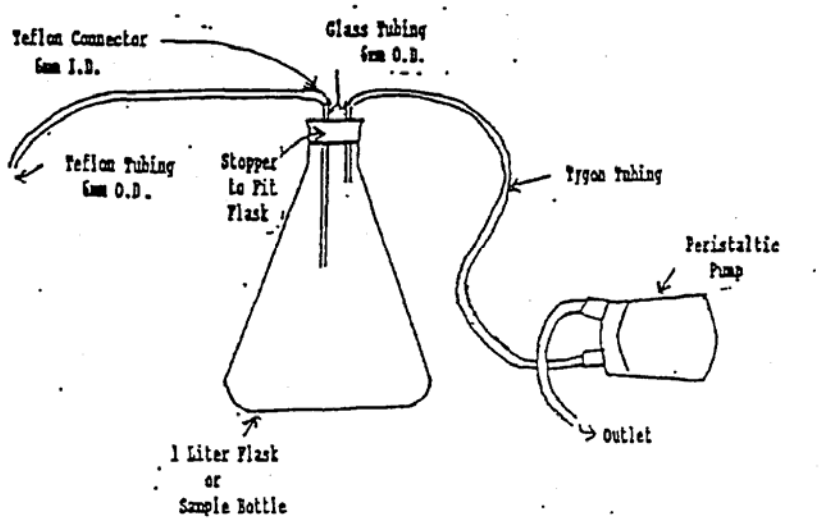
Samples from shallow depths can be readily collected by submerging the sample container. The method is advantageous when the sample might be altered during transfer from a collection vessel into another container. This is the case with samples collected for oil and grease analysis because considerable material in the surface film may adhere to the sample transfer container and as a result produce inaccurately low analytical results. Similarly the transfer of a liquid into a small sample container for volatile organic analysis, if not done carefully, could result in significant aeration and resultant loss of volatile species. If the water is considered to be hazardous, the external surface of each container may need to be decontaminated. Another disadvantage with this method is that the water surface is disturbed at least once for each sample, whereas the use of a larger, transfer sampler will disturb it fewer times.

It is often necessary to collect liquid samples at some distance from shore or the edge of the containment. In this case, a useful device is the pond sampler (deVera et al., 1980) which incorporates a telescoping heavy-duty aluminum pole with an adjustable beaker clamp attached to the end. A beaker or other disposable glass or plastic container, or the

actual sample container itself, can be fitted into the clamp. In situations where cross contamination is of concern, use of a disposable container or the actual sample container is always advantageous. The cost of proper cleaning usually outweighs the cost of disposal of otherwise reusable glassware or bottles. This is especially true when the cleanup must be done in the field. The potential contamination of samples for volatile organic analysis by the mere presence of organic solvents necessary for proper field cleaning (see Section 3.2.4) is usually too great to risk.

Another method of extending one's reach in collecting samples for analyses of nonvolatile organics is the use of a small peristaltic pump. In this method the sample is drawn in through heavy-wall Teflon tubing and pumped directly into the sample container. This system allows the operator to reach out into the liquid body, sample from depth, or sweep the width of narrow streams. Because the interior of the peristaltic pump requires flexible tubing and most flexible tubing is not inert to many hazardous wastes, a vacuum flask can be inserted in line before the pump assembly as shown in Figure 3.2-1. A peristaltic pump can be used to collect samples at depths up to several meters in ponds or other containment vessels. Peristaltic pumps are not recommended for the collection of samples for volatile analysis.

Figure 3.2-1 Peristaltic Pump Sampler (from National Council of the Paper Industry for Air and Stream Improvement, Inc., 1982)



In situations in which samples are required from depths greater than the capabilities of a peristaltic pump (approximately 25 feet), samplers such as Kemmerer, ASTM Bomb (Bacon Bomb), or Van Dorn samplers can be used; however, care must be used in selecting devices that are made of materials that will not contaminate the sample. See Ford et al. (1984) for more details on these samplers. Coliwasa samplers can also be used for surface water samples, especially where uniform samples with depth are required. These are described in Section 3.3, Industrial Waste Sampling. Determination of the locations and depths from which samples should be collected and the appropriate number of samples is discussed in Section 3.1.2.

3.2.4 Decontamination of Equipment.

Laboratory decontamination of sampling equipment is preferable to field decontamination but is not always possible. In general, when a piece of equipment must be cleaned in the field and reused, it should only be used to collect samples expected to be more highly contaminated. It must never be used if it appears discolored or otherwise obviously contaminated.

Nondisposable pumps should be cleaned in the field by pumping a solution of non-phosphate detergent through the pump and associated tubing. This solution should be followed by tap water, then followed by purified water.

Nondisposable bailers should be disassembled and cleaned by washing in non-phosphate detergent, followed by rinses with tap water and deionized water. They should then be air

dried in a clean environment, reassembled using gloves, and wrapped or sealed in a clean plastic bag. When organic compounds are of concern, isopropyl alcohol (rubbing alcohol) should be considered for decontamination, since it has the advantage of drying wet surfaces quickly, dissolving many organic compounds, and being less toxic and less flammable than other solvents.

Filtering apparatus should be of a disposable variety eliminating the decontamination process. In those rare instances filtering equipment is reused, it should be cleaned in a solution of a non-phosphate detergent, followed by rinses with tap water, a dilute nitric acid solution, and finally, deionized water.

Equipment blanks should be collected from all equipment cleaned in the field and reused, to detect any contamination not removed by or introduced by the cleaning procedure.

When equipment is returned to the laboratory or office, it should be thoroughly cleaned. All waste decontamination fluids and materials should be collected and properly disposed.

3.2.5 Quality Control for Water Sampling.

Quality control samples should be collected according to procedures described in Section 5. These procedures are described below more fully as they apply to water samples.

Collocated samples should represent 5% of the samples collected. Usually this means that a second set of samples is taken from one well. This well should be named in the sampling plan, i.e., chosen before sampling begins.

Split samples are more difficult to obtain, because they involve collecting enough sample in an intermediate container to decant into all sample bottles for that analysis. For analysis of volatile components, the sample should be handled as little as possible to minimize loss. VOA bottles should be filled with water from the same bailer, if possible. If the bailer does not hold enough water to fill all VOA bottles needed, then water from each bailer filled should be distributed among all VOA bottles. Field notes should contain information on how the sample was split.

Both collocated and split samples are useful replicate samples to collect. Split samples are used to determine the precision or reproducibility of the analyses. They are especially useful for interlaboratory comparisons. But because of the possibility of loss of constituents during the splitting process, collocated samples may be more accurately reflect in-situ concentrations. These samples may also yield a measure of error from the sampling procedures.

Travel blanks, consisting of distilled water from the laboratory, are used mainly for volatile analyses. They should be brought out to the field in sample containers and returned to the laboratory, one with each cooler, to account for any contamination which may occur from container handling.

Equipment blanks should be taken each day, using distilled water and passing it through each equipment procedure used. Bailers or pump assemblies should have distilled water collected as blanks, filtration units should be sampled, and all preservatives used in the field should be included.

Additional quality control samples are described in Section 5.0.

3.2.6 References.

1) The California Environmental Protection Agency, July 1995. Representative Sampling of Ground Water for Hazardous Substances, Guidance Manual for Ground Water Investigations.
2) Nielsen, David M., 1991. Practical Handbook of Ground-Water Monitoring. Lewis Publishers, Inc.

3) Puls, Robert W., and Barcelona, Michael J., April 1996. Low-Flow (Minimal Drawdown) Ground-Water Sampling Procedures. United States Environmental Protection Agency, EPA/540/S-95/504.

4) Geological Services Unit, Geology and Corrective Action Branch, Department of Toxic Substances Control, July 2001. Guidance Document, Monitoring Requirements for Permitted Hazardous Waste Facilities, Appendix B.

3.3 INDUSTRIAL WASTE SAMPLING.

This section covers sampling and analysis for industrial wastes. The discussion of health and safety plans for such activities, although of critical importance, is not included here. The intent is to address the sampling of process wastes, as may occur during facility inspections or site investigations. Future revisions of SW-846 Chapter 10 will provide additional guidance on sampling techniques.

3.3.1 General Considerations.

In contrast with groundwater sampling, which was discussed in the previous section, industrial waste sampling generally involves concentrated liquids, solids, slurries, or sludges. The choice of sampling technique depends on the physical state and the chemical composition of the waste. The Field Manual of SW-846 and the EPA Manual "Samplers and Sampling Procedures for Hazardous Waste Streams" provide some guidance in sampling techniques. In addition, the American Society for Testing and Materials (ASTM) has published numerous Standard Methods for sampling commercial products and wastes. For waste piles, reference ASTM D 6009-96 titled "Standard Guide for Sampling Waste Piles". This standard may be obtained from ASTM through their web site: http://www.astm.org

3.3.2 Material Compatibility.

Because of the concentrated nature of the waste, chemical compatibility is an important issue in the choice of sampler. In general, glass or Teflon are acceptable materials for sampling of concentrated wastes. Table 3.3-1 from SW-846 lists some acceptable samplers for given sampling situations.

3.3.3 Choice of Sampling Technique.

As is obvious from Table 3.3-1, the choice of sampling technique will be determined by the waste container. In most cases, the goal is to obtain a sample which is representative of the waste unit. For example, drum sampling should produce samples which include all layers in the drum, since drummed material is often stratified. Sampling techniques may need to be modified for particular sampling situations. The sampling of a large number of drums of unknown material may indicate that sampling with disposable glass thieves is preferable to sampling with a reusable Coliwasa, due to potential chemical incompatibility and difficulties in cleaning samplers between samples. Disposable glass Coliwasa are also available commercially (e.g., from Pollution Abatement Consultants and Services, P.O. Box 1039, Millville, New Jersey 08332) and can eliminate some of the problems of cross-contamination and sampler cleaning.

Sampling of unknown industrial waste should be done simultaneously with some field screening. This serves two purposes: first, it guides sampling by identifying wastes with similar properties, and allows for a refinement of sampling plans. Second, it can identify waste properties and guide subsequent lab analysis requests. Field screening can also identify potential problems which should be addressed in the field, such as the discovery of water-reactive wastes. All pertinent field information on waste characteristics should be transmitted to the lab which will be doing the analysis. Table 3.3-2 lists field measurements and the corresponding lab analysis for a variety of chemicals.

Due to the potential for cross-contamination, dedicated sampling equipment should be used for sampling different waste types when feasible. If samplers are to be used repeatedly, clean the equipment with appropriate solvents. The choice of cleaning solvents depends on the waste and the expected chemical analysis. For the sampling of unknown organics, isopropanol rinses are recommended. When doubt exists as to the cleanliness of sampling equipment, a solvent rinse (equipment blank) can be submitted to the lab along with samples for chemical analysis.

Highly stratified heterogeneous wastes, e.g., globules of oil mixed with soil, should be sampled by collecting samples of each stratum. This is because

1) conventional sampling techniques may not be effective, and

2) an average value for the waste would not be valuable in predicting the properties of individual samples.

The American Society for Testing and Materials (ASTM) has published a standard guide on Sampling strategies for heterogeneous Wastes which can be found in the References at the end of this section.

	Waste Location or Container								
WASTE TYPE	DRUM	SACKS AND BAGS	OPEN-BED TRUCK	CLOSED-BED TRUCK	STORAGE TANKS OR BINS	WASTE PILES	PONDS, LAGOONS, AND PITS	CONVEYER BELT	PIPE
FREE-FLOWING LIQUIDS AND SLURRIES	Coliwasa	N/A	N/A	Coliwasa	Weighted Bottle	N/A	Dipper	N/A	Dipper
SLUDGES	Trier	N/A	Trier	Trier	Trier	a	a		
MOIST POWDERS OR GRANULES	Trier	Trier	Trier	Trier	Trier	Trier	Trier	Shovel	Dipper
DRY POWDERS OR GRANULES	Thief	Thief	Thief	Thief	a	Thief	Thief	Shovel	Dipper
SAND OR PACKED POWDERS OR GRANULES	Auger	Auger	Auger	Auger	Thief	Thief	a	Dipper	Dipper
LARGE-GRAINED SOLIDS	Large Trier	Large Trier	Large Trier	Large Trier	Large Trier	Large Trier	Large Trier	Trier	Dipper

[a] This type of sampling situation can present significant logistical sampling problems, and sampling equipment must be specifically selected or designed based on site and waste conditions. No general statement about appropriate sampling equipment can be made.

Table 3.3-1

AVAILABLE INFORMATION	ANALYTICAL REQUEST
Suspect high levels of volatile organics (high field readings on explosivity meter, OVA, or PID)	Headspace VOA and Flash Point. (If VOA results show organics of concern, a request should be submitted for specific analyses, e.g., purgeable halocarbons, purgeable aromatics, etc.)
Positive CLOR-N-OIL™	Chlorinated pesticides and PCBs, TOX
High (>1,000 ppm) CLOR-D-TECT™ result	Total halogens
Positive Enzytec ET-10 test	Organophosphates and/or carbamates
Positive PCB immunoassay	PCBs
Positive Beilstein test	Chlorinated pesticides and PCBs
Elevated radioactivity	Gross alpha and Gross Beta radioactivity
Extreme field pH (<3 or >11)	pH
High field sulfide test result	Total sulfides
High field cyanide test result	Total cyanides
Suspect high levels of toxic metals	Metals, WET if necessary

Table 3.3-2. Guide to Requesting Analysis

3.3.4 References

1) ASTM Standard Guide on Sampling Strategies for Heterogeneous Wastes, D5956-96

3) U.S.E.P.A., Characterizing Heterogeneous Wastes, EPA 600/R-92/033, February, 1992

3.3.5 Wipe Sampling.

3.3.5.1 Application.

In some situations, it is necessary to measure the contamination of a surface. Surface contamination measurement may be necessary to:

Verify equipment decontamination, e.g., as part of a clean closure.

Estimate the level of particulate fallout, e.g., from dust fall.

Determine the extent of residual contamination of PCBs after a clean-up.

3.3.5.2 Wipe Procedure

SW-846 does not contain a standard wipe sampling procedure, but the field manual of the Occupational Health and Safety Administration (OSHA), 1990, does specify a procedure to be followed. Some details are shown in that manual. EPA has published a procedure in regulation for wipe sampling following a PCB spill.

The factors to consider in wipe sampling are the wipe material, the solvent, the sampling area, and the use of blanks.

The solvent applied to the wipe material will depend on the contamination being investigated. For PCBs, chlorinated solvents, or semi-volatile organics, a glass fiber filter (37 mm) wetted with hexane or another organic solvent is recommended. Paper filters moistened with acidified deionized water may be used for metals and other analysis.

The area for the wipe sample should be carefully measured and the margin for error estimated. Typically, a 10 cm X 10 cm (100 cm^2) area is wiped. For quality control, replicate wipe samples should be taken from the same general area. The wipes should be placed in containers as described in Section 3.6.

Field blanks should always be used. These would be wipes moistened with the same solvent and placed in the same type of container as the samples. A field blank should be submitted to the laboratory with each batch of wipe samples.

3.3.5.3 Interpretation

The lab results for a wipe sample should be in units of mass, e.g., mg or ug per wipe sample. The actual proportion of contaminant recovered by wiping with a solvent cannot be measured, although studies have shown that 80% of a lead contamination can be recovered from a smooth surface (Chavalitnitikal, 1984).

The surface concentration can then be calculated using the known surface area of the wipe and converting to units of mass per unit area, e.g., mg/m^2. If results in units of

concentration, e.g., mg/kg, are needed, then the lab must provide pre-weighed (tared) wipes.

3.3.5.4 References

1) Chevalitnitikal, Chaiyuth A., "A laboratory evaluation of wipe testing based on Lead Oxide Surface Contamination," Am Ind Hyg Assoc J, 45(5), pp 311-317, 1984.

2) EPA, 40CFR "Subpart G-PCB Spill Cleanup Policy," 761.123 Definitions.

3) OSHA, "Chapter 2; Sampling for Surface Contamination," Volume VI - OSHA Technical Manual, March 26, 1990.

3.4 SOIL SAMPLING.

The following discussion gives a brief description of surface and near surface soil sampling primarily based on the references listed in subsection 3.4.5. The reader should consult those references for more detailed information.

3.4.1 General Considerations.

The selection of sampling techniques and sampling devices for soils should take into account the following points:

1) The objectives of the sampling effort which will determine the number of samples to be collected, the required sampling depth and whether or not the samples are to be composited. See Section 3.1 for additional discussion of sampling plans.

2) The physical properties of the soil, e.g., grain size, cohesiveness, homogeneity and presence of anomalies, such as animal burrows, large rocks or plant roots. Certain samplers which work well with soft, fine-grained soils may not work with hard, rocky soils.

3) Thickness of the soil layer above the bedrock or water table which may limit the depth from which samples can be collected.

4) The amount of sample required. The minimum sample size is specified by the laboratory on the basis of the analytical method and the required sensitivity of analysis.

5) The type(s) of elements or compounds for which the samples will be analyzed. This consideration may preclude the use of samplers made of certain materials (e.g., certain metals, PVC).

Samples should not be collected immediately after heavy rainfall, when the soil is frozen, or in extreme winds (Barth and Mason, 1984b).

3.4.2 Preparation of the Sampling Site.

If the soil layer at a given sampling site is covered with vegetation or other non-soil matter, any such soil cover is removed using a spade, shovel or scoop. The same precautions with respect to structural materials described for samplers in section 3.4.4.1. must be taken with these tools.

An area large enough to collect all samples to be taken at the site (e.g., for subsequent compositing) should be cleared before beginning to sample.

3.4.3 Sample Compositing.

When the major concern of the sampling effort is to establish the distribution of contaminants between different soil horizons or their paths of migration within the soil layer, the collected samples should not be composited.

When the major concern is to obtain a sample which is representative for a particular site, it is recommended that four or more different samples taken at the site be composited into a single sample (Barth and Mason, 1984b). Equal amounts of the different samples should be used for compositing.

NOTE: Compositing effectively raises the detection limits for all potential contaminants at the site because localized high levels of contamination are diluted by mixing with relatively uncontaminated soil.

3.4.4 Sampling Devices.

3.4.4.1 Sampler Materials.

Most commercially available samplers for soil are made of steel, brass or plastic. When gardening tools, such as spades, shovels or trowels are employed, implements made of nickel- or chromium- plated steel should be avoided since the coating may flake off and severely affect the results of trace element analyses.

Painted surfaces are even more subject to abrasion and paint may interfere with determinations of organics and/or trace metals. Plated or painted implements can be used in many cases, however, if the surface coating is removed, e.g., by sandblasting (Ford et al., 1984).

Plastic materials are frequently used as liners of coring samplers; while such liners may be appropriate for soils needing metal analyses, plastic materials are usually not suitable when samples are collected for organic analysis.

Because of the above considerations, it is generally advisable to use samplers which are made of as few materials as possible in order to reduce the number of potential contamination sources. The possibility of contamination from plasticizers should always be considered.

3.4.4.2 Sampler Types.

This section is limited to sampling devices which can be employed with a minimum of special training, equipment or cost. Depending on the physical properties of the soil, sampling depths are limited to 5 meters (Veihmeyer sampler). Sampling to greater depths and under difficult soil conditions usually require drilling equipment and experienced drilling personnel.
An overview hand operated sampling devices for different sampling depths are given in

Table 3.4-1. Detailed descriptions of the use of these samplers can be found in the references indicated in the table.

Ford and coworkers (1984) have given good concise descriptions of a method for near-surface sampling using a spade and scoop and a method for sampling at intermediate depths using an auger and thin-wall tube samplers.

3.4.4.3 Sampler Cleaning and Decontamination.

In order to minimize the contamination of soil samples by the sampling equipment or through cross-contamination, all equipment must be thoroughly cleaned before their first use and also between samples.

The following is a suggested procedure that have been used effectively at the field sites. (Reference 5)

1. Wash and scrub tools with tap water using pressure hose or pressurized stainless steel, fruit tree sprayer. If necessary use a steel brush or other brush to remove adhered soil such as sticky clays. A steam cleaner has been proven to be very effective at this step in the cleaning operation.

2. If organics are present, rinse with the waste solvents from the steps outlined below. Discard contaminated solvent by pouring into a waste container for later disposal.

3. Air dry the equipment or dry with acetone.

4. Double rinse with distilled water.

5. If organic pollutants are of concern, rinse with spectrographic grade acetone saving the solvent for use in step 3 above.

6. Rinse twice in spectrophic grade hexane, saving the solvent for used in step 3 above. Methanol can be used if proper precautions are taken.

7. Air dry the equipment.

8. Package in plastic bags and/or pre-cleaned aluminum foil.

9. Collect contamination blanks to insure that sampling equipments are properly cleaned.

Many disposable samplers are available which minimize the possibility of cross-contamination.

SAMPLING DEPTH	SAMPLER TYPE	APPLICATION	REFERENCES
Near Surface (0-6 inches)	Trowel or Scoop	Top 10 cm only	2,3
	Shovel or Spade	Wide variety of soil condition	3
Mid-Depth (0-72)inches	Trier	Difficult to use with hard, rocky soils	1,2
	Ring lined sampler	Cohesiveness, wet to medium soil	5
	Soil probe or King-tube	cohesive, soft soils; representative samples in soft to medium cohesive soil & silts	6
	Thin-walled tubes	cohesive, soft soils; special tips for wet or dry soils	6
	Soil Recovery Probe	Cohesive soft soils; cores are collected in reusable liners	6
	Peat Sampler	Wet, fibrous, organic soils	6
	Screw Bucket Auger	Cohesive, soft or hard soils	6
	Standard Bucket Auger	General soil	6
	Sand Bucket Auger	Retain dry, loose or granular material (silt, sand & gravel)	6
	Mud Bucket Auger	Wet silt and clay soil	6
	Dutch Auger	Wet, fibrous or rooted soils	6
	In Situ Soil Recovery Auger	Collection of soil samples in reusable liners	6
	Eijkelcamp Stoney Soil Auger	Stoney soils and asphalt	6
	Planer Auger	Used to clean out and flatten the bottom of predrilled holes	6
	Post-Hole/Iwan Auger	Cohesive, soft, or hard soils	6
	Silage Auger	Silage pits and peat bogs	6
	Spiral Auger	Removes rock from auger holes	6
Deep Samples (>72 inches)	Veihmeyer sampler	Cohesive soil to depth of 3 meters (118 inches)	2, 6

Table 3.4-1: Overview of Hand operated soil sampling devices.

3.4.4.4 Soil Sampling for Volatile Organics

Because of the potential to lose volatiles during sampling, storage, and subsampling in the laboratory, special sampling procedures are needed for volatile organics in soil.
The tube type samplers such as Shelby tubes and split spoon samplers can be used for initial sampling for volatile organics. These samplers can collect a relatively undisturbed, intact soil sample; a sub-sample should be taken immediately. For high level (<200 ug/kg) analysis, a sample is taken with a hermetically sealed sampler or preserved with methanol. For low level analysis, the sample is taken in a hermetically sealed sampler or transferred into a VOA vial with sodium bisulfate, water, and a stirring bar, which can be used directly in purge-and-trap analysis, as described in EPA Method 5035.

3.4.5 References.

1) Barth, D.S. and B.J. Mason. 1984a. Soil Sampling Quality Assurance Guide. EPA-600/4-84-043

2) Barth, D.S. and B.J. Mason. 1984b. Soil Sampling Quality Assurance and the Importance of an Exploratory Study. In: Environmental Sampling for Hazardous Wastes, G.E. Schweitzer and J.A. Santolucito, eds., ACS Symposium Series 267, ACS, Washington, D.C.

3) Mason, B.J. 1983. Preparation of Soil Sampling Protocol: Techniques and Strategies. EPA-600/4-83-020. Environmental Monitoring Systems Laboratory, U.S. EPA, Las Vegas, NV.

4) EPA, "Behavior and Determination of Volatile Organic Compounds in Soil: A Literature Review," EPA 600/R-93/140, EMSL-Las Vegas, May, 1993.

5) Mason, B. J. Preparation of Soil Sampling Protocols: Sampling Technique and Strategies. EPA/600R-92/128, EMSL-Las Vegas, July 1992

6) EPA, "Description and Sampling of Contaminated Soils", EPA/625/12-91/002, November 1991.

3.5 SOIL GAS.

Soil gas measurements can be extremely important in evaluating site contamination. Volatile organics will partition among soil, water, and soil gas depending on their volatility, water solubility, and affinity for soil.

Information on soil gas is useful in defining soil contamination related to: land-filled hazardous materials; contaminated ground water including leachate plumes from landfills; and leaking underground fuel tanks.

Soil gas sampling is particularly difficult to do accurately because in making the measurement we disturb the soil and soil structure. Since we expose fresh soil surfaces to the atmosphere, steep gas concentration gradients develop and the soil rapidly "off-gases".

The discussion provided here outlines methods which have been used, without making specific recommendations. More detailed protocols have been published by the Los Angeles Regional Water Quality Control Board (Ref 1) and the U.S. EPA Emergency Response Team (Ref 2).

Several new passive and active soil gas sampling techniques have been developed in conjunction with push technologies for sub-surface investigations.

3.5.1 Bore Hole Gas Probe Sampling Technique.

Soil gas can be sampled at various depths in the soil profile using bore hole techniques. One procedure which has proven useful is the bore hole probe technique in which a 2-4 inch diameter hole is made by augering to the depth of interest. A hollow probe with a perforated tip is then driven about one foot into the soil at the bottom of the hole allowing soil gas at that depth to be pumped to the surface with a peristaltic pump. The gas sample is then collected in a gas collection bottle located between the probe and the pump. A flow meter is needed since it is important that the pumping rate does not exceed 10-100 mL/minute. Excessive flow rates create a partial vacuum and draw clean air into the soil, diluting the sample. A problem occasionally encountered is ground water, which if reached will be sucked into the gas collection bottle and contaminate the sampling device.

A small hollow stem auger (6" in diameter with a 3" inside diameter) can be useful for depth specific sampling at depths too great for a hand auger. Drilling is stopped at specified depths and sampled by the bore hole technique. (This procedure requires a cooperative drill rig operator.)

3.5.2 Sealed Bore Hole Technique.

The sealed bore hole technique does not allow soil gas to be sampled at various depths in the soil profile, but rather gives a depth-averaged soil gas sample. This technique usually gives sufficient information for a preliminary characterization of soil gas at the site. There are several variations to the sealed bore hole technique. One is to auger a 6-12" deep hole and insert a 3/4" in diameter Teflon^{R} probe with holes drilled at the tip to avoid plugging. The probe is attached by various fittings to a peristaltic pump as in Section 3.5.1.

For sampling at greater depths, insert the sampling probe into a 3-5 foot deep bore hole (about 2" in diameter) and pack topsoil around the probe. An aluminum foil plate with a hole for the sampling tube is placed at the top of the sample hole and covered with soil to seal the chamber. The sampling probe should be extended about half way down the bore hole. Allow 24-48 hours for the soil gas in the auger cavity to reach equilibrium with the surrounding soil and sample as in Section 3.5.1.

A simple technique for sampling uses a small (3/8") diameter steel rod to create an open hole at the desired depth. The rod is removed and a 1/4" diameter stainless steel probe is inserted into the open hole. Topsoil is packed around the tube to seal the system from ambient air. The gas can then be sampled as in Section 3.5.1. or drawn into a Tedlar bag located in a vacuum desiccator The small rod is easier to drive and allows sampling in otherwise inaccessible areas.

3.5.3 Headspace Technique.

This technique is the most difficult because of major losses of the most volatile constituents. As a result, this technique may be most valuable for qualitative rather than quantitative measurements. The sample can be fully characterized by GC/MS in the laboratory, but the concentrations reported are likely to underestimate the most volatile contaminants.

In the headspace technique, sufficient subsurface soil is collected in a glass jar with a Teflon lined septum to fill the jar to at least ½ the total volume. The jar is then transported to the laboratory and the headspace is sampled directly by syringe.

3.5.4 Solid Sorbent Sampling Technique.

This technique adsorbs the soil gases on a solid matrix (i.e., tubes containing Tenax-GC, activated charcoal, or XAD-2) in the field and then desorbs the sample in the laboratory. The soil gas can either be pumped across the sorbents or allowed to diffuse passively over a period of time. Low flow monitoring pumps must be capable of maintaining consistent flows at prescribed rates. Characteristics of the compound(s) and sorbent of interest should be investigated thoroughly before attempting this procedure. For example, the optimum volume of gas and rate of pumping will depend on target compounds, sorbent, detection limits, and expected concentrations.

3.5.5 Construction of Samplers.

Samplers should be constructed of stainless steel or Teflon. Tygon, rubber, and other types of tubing are to be avoided because they may give inaccurate results due to adsorption and release of volatiles. Gases adsorb and desorb from Teflon to a certain extent, and for this reason stainless steel is preferable.

3.5.6 Cleaning of Sampling Equipment.

The sampling device should be cleaned with a detergent, rinsed with deionized water, and dried in a clean environment before performing any field work. If necessary, rinse the sampling device with deionized water between samples, followed by purging (blowing out) with hydrocarbon free air. Pumping ambient air through the system must be verified as an effective means of cleaning.

3.5.7 Quality Control.

Quality control samples should include field blanks (ambient air or hydrocarbon free air) to check for sample carryover, duplicates taken from the same bore hole, a standard reference gas if available, and spiked samples when appropriate.

3.5.8 Portable Vapor Detectors.

There are various commercially available portable direct reading vapor detectors (PVD) which are useful for rapid field evaluation. The photoionization detector (PID) is a vapor detector which has adequate sensitivity for organics like benzene (0.5 ppm) and halogenated solvents, but does not detect methane. Its use is very straightforward-- to evaluate soil gas a 12" hole is augered and the PID probe is inserted and read. Only highly contaminated sites can use this method. Another useful PVD is the Century OVA 128 (with a flame ionization detector, FID) which, unlike the PID, responds to all volatile organics about equally. The OVA can be operated in continuous, total, or GC modes and thus has the ability to differentiate some components of the gas mixture.

The PVDs are most useful for rapid, preliminary site assessments. Typical applications include:

1) Location of "hot" spots.
2) Crude definition of depth profile (for example, surface spills can be distinguished from land-filled barrels).
3) Screening wells for contamination.
4) Field guidance to crews drilling wells.
5) Protecting the health and safety of field staff.

3.5.9 Soil Gas References

1. California Regional Water Quality Control Board, Los Angeles Region, "Requirements for Active Soil Gas Investigation, Well Investigation Program," March, 1994.

2. U.S. EPA Office of Solid Waste and Emergency Response, "Compendium of ERT Soil Sampling and Surface Geophysics Procedures," EPA/540/P-91 006, January, 1991.

3.6 CONTAINERS.

Specific containers are required for some tests. Generally, samples for organic analysis are collected in glass containers. Glass containers are free of organic plasticizers. Certain organics which are sensitive to light and decompose easily are collected in amber glass containers. Samples for inorganic (metals and anions) analysis are generally collected in plastic or glass containers.

3.6.1 Sample Containers.

The Sampling and Analysis Plans should identify the type of containers to be used for sample collection. When none are specified, the following guidelines will apply:

Type:

When organics are the analytes of interest, glass bottles with Teflon-lined caps should be used. When metals are the analytes of interest, polyethylene containers with polypropylene or polyethylene caps should be used. Glass bottles with Teflon-lined caps may also be used for metals.

Several common container types are listed on the reverse side of the Sample Analysis Request form (fig. 4.0-4).

Size:

1) For soils and wastes, 8 oz. wide-mouth jars with Teflon closures should generally be used. Filling the container half full will provide sufficient sample for most analyses. However, for soils and wastes requiring volatile organic analysis, a 4 oz. wide-mouth jar (filled to capacity to minimize headspace) will suffice. See also Table 3.7-2 in Section 3.7.

2) Containers for water samples are specified in Table 3.7-3 in Section 3.7.

3.6.2. Container Cleaning.

Containers may be purchased pre-cleaned and certified and are preferred to in-house cleaning. These containers may be purchased from the following sources:

I-Chem Research Inc.
2 Bouilen Circle, Suite 8
New Castle, DE 19720
1-(800) 262-5006

Scientific Specialties Service
P.O. Box 352
Randalstown, MD 21133
1-(800) 648-7800

Eagle Picher
36 BJ Tunnel Blvd.
Miami, OH 74354
1-(800) 331-7425

Environmental Sampling Supply
9601 San Leandro Blvd.
Oakland, CA 94603
1-(800) 233-8425
1-(510) 562-4988

When pre-cleaned and certified containers are not available, the containers should be cleaned to suit the type of analysis required. When samples are to be analyzed for metals (e.g. Method 6010, SW-846), the cleaning procedure for containers and closures should be:

1) Thoroughly wash with non-phosphate detergent (e.g. Liquinox) and hot tap water.
2) Rinse three times with tap water.
3) Rinse with nitric acid (1:1).
4) Rinse three times with ASTM Type II water.
5) Glass, oven dry; plastic, air dry.

When samples are to be analyzed for organics glass containers are required. The cleaning procedure for the containers and closures should be:

1) Thoroughly wash with non-phosphate detergent and hot tap water.
2) Rinse three times with tap water.
3) Rinse with nitric acid (1:1).
4) Rinse three times with ASTM Type II water.
5) Rinse with methylene chloride.
6) Oven dry.
7) Bake at 400°C. (when required).

Other cleaning procedures may be used when required by the Sampling and Analysis Plan. For example, it may be necessary to delete the use of methylene chloride when analyzing for volatile organics.

A modified procedure may be used if it can be documented through an active analytical quality control program using spiked samples and field blanks that the procedure is adequate for its intended use.

3.7 SAMPLE PRESERVATION.

3.7.1 Preservation.

The Sampling and Analysis plan should identify sample preservation methods that are to be used. Where none are specified, the requirements given below should be used.

Methods of sample preservation are relatively limited and are generally intended to 1) retard biological action, 2) retard hydrolysis, and 3) reduce absorption effects. Preservation methods are generally limited to pH control, chemical addition, refrigeration (4°C.), and freezing (-15°C.).

3.7.2 Preservation of Soil and Waste Samples.

Preservation of soil and wastes is generally limited to refrigeration. High level samples for semivolatile and metal analysis (those with analyte concentrations above 1%) generally do not require preservation. Low level (<10 mg/kg) to mid level (10 to 10,000 mg/kg) samples generally require preservation by cooling to 4°C. See Table 3.7-2.

3.7.3 Preservation of Water Samples

Preservation for water samples is given in Table 3.7-3.

Table 3.7-2 Sampling and Preservation for Soil and Wastes

PARAMETER	CONTAINER[a]	PRESERVATION	HOLDING TIME	SAMPLE SIZE
Acidity	P,G	Cool, 4°C		4 oz jar
Alkalinity	P,G	Cool, 4°C		4 oz jar
Chloride	P,G	None		4 oz jar
Chromium VI	P,G	Cool, 4°C	30 days (EPA 3060A)	4 oz jar (fill to minimize headspace)
Conductivity	P,G	Cool, 4°C		4 oz jar
Cyanide (Total & amenable to chlorination)	P,G	Cool, 4°C	14 days	4 oz jar (fill to minimize headspace)
Cyanide (Reactive)	P,G	Cool, 4°C store in dark	Analyze as soon as possible	4 oz jar (fill to minimize headspace)
Fluoride	P,G	None		4 oz jar
Nitrate	P,G	Cool, 4°C		4 oz jar
Organolead	G-Amber	Cool, 4°C	14 days	4 oz amber glass (fill to minimize headspace)
pH	P,G	None	Analyze as soon as possible	4 oz jar
Sulfate	P,G	Cool, 4°C		4 oz jar
Sulfide (Total)	P,G	Cool, 4°C Fill surface of solid with 2N Zn acetate until moistened	Analyze as soon as possible	4 oz jar (fill to minimize headspace)
Sulfide (Reactive)	P,G	Cool, 4°C store in dark	Analyze as soon as possible	4 oz jar (fill to minimize headspace)
Extraction Procedure Toxicity	Glass, Teflon-lined Septum	Do not add preservative. Refrigerate only if sample integrity is not affected		1 L for liquid sample, 200 g for solid sample
TCLP	Glass, Teflon-lined Septum	same as above	Vol & semi-vol-14 days Hg-28 days Metals (except Hg)-180 days, see footnote d	Liquid sample: 1 L minimum for each category of analysis; Solid sample: 250 g
Total Phosphate	P,G	Cool, 4°C		4 oz jar

Table 3.7-2 Sampling and Preservation Requirements for Soil and Wastes (continued)

PARAMETER	CONTAINER[a]	PRESERVATION	HOLDING TIME	SAMPLE SIZE
Total Metals (except Cr VI and Hg)	P,G	None	6 months (except Hg, 28 days)	4 oz jar
Volatile Organics: soil, sediments & sludges	Single transfer sampler (eg. Encore ™) **For field preservation call ECL**	Cool, 4°C.	48 hours Note: High level preserved in MeOH within 48 hours extends to 14 days	low level-5 g size<200 ug/Kg high level-25 g size >200 ug/Kg
Volatile Organics: concentrated wastes	G	Cool, 4°C.	14 days	4 oz jar (fill to minimize headspace)
Semivolatile Organics: soil, sediments & sludges	G	Cool, 4°C	14 days to extract; 40 days to anal. after extraction.	8 oz widemouth jar (1/2 full)
Semivolatile Organics: concentrated wastes	G	None	14 days to extract; 40 days to anal. after extraction.	8 oz widemouth jar (1/2 full)
PCDD/PCDF	G	Cool, 4°C	1 year to extract; 40 days to anal. after extraction	8 oz widemouth jar (1/2 full)

[a] P =Polyethylene container with polypropylene closure.
G = Glass container with Teflon-lined closure.
G-V = Glass VOA vial or bottle with Teflon septum

[b] Minimum volume for analysis shown in ( ).
For VOA samples intended to be submitted to the laboratory in end-capped core tubes, there is evidence that preserving by freezing with dry-ice is superior to preserving by cooling to 4°C. Contact ECL for details.
Ref.: Data from P. King, P & D Environmental.

[c] There is evidence that VOA soil samples preserved in methanol during the field sampling and cooled to 4°C is superior to simply preserving by cooling to 4°C. However, the methanol used for preservation must be absolutely pure in order to avoid introducing volatile contaminants. A field blank is also required. Contact the laboratory for preservation details and sample handling procedure.
Ref.: *Environ. Sci. Technol.*, **1990**, 24, 1387-1392.

[d] Maximum sample holding times from field collection to TCLP extraction. See Method 1311 for the preparative and analysis holding times for TCLP extracts.

Table 3.7-3 Sampling and Preservation for Water and Wastewater

PARAMETER	CONTAINER[a]	PRESERVATION	HOLDING TIME	SAMPLE VOL, mL(minimum)[b]
Acidity	P,G	Cool, 4°C	14 days	100 (100)
Alkalinity	P,G	Cool, 4°C	14 days	100 (100)
Ammonia	P,G	Cool, 4°C H_2SO_4 to pH <2	28 days	1000 (500)
Asbestos	P	Cool, 4°C	48 hours	1000 (1000)
Boron	P,G	none	28 days	100
Chloride	P,G	none	28 days	100 (50)
Chromium VI	P,G	Cool, 4°C	24 hours	500 (200)
Conductivity	P,G	Cool, 4°C	24 hours	100 (50)
Cyanide (Total & amenable to chlorination)	P,G	Cool, 4^{o} C, 10 N NaOH to pH>12; 0.6g Ascorbic acid/L if Cl present or 5 ml 0.1 N sodium arsenite/L	14 days	1000 (500)
Cyanide (Reactive)	P,G	May adjust to pH 12 with NaOH but sample integrity maybe affected. Cool, store in the dark.	Analyze as soon as possible	100 (100), fully filled with zero headspace.
Hardness, Total	P,G	HNO_3 to pH <2	6 months	100 (50)
Fluoride	P,G	none	28 days	300 (100)
METALS (except Cr VI and Hg):				
Dissolved	P,G	Filter onsite HNO_3 to pH <2	6 months (except Hg, 28 days in glass, 13 days in plastic)	1000 (500)
Total	P,G	HNO_3 to pH <2	6 months (except Hg, 28 days in glass, 13 days in plastic)	1000 (500)
Nitrate	P,G	Cool, 4°C	48 hours	50 (50)
Nitrite	P,G	Cool, 4°C	48 hours	50 (50)

Table 3.7-3 Sampling and Preservation for Water and Wastewater (Continued).

PARAMETER	CONTAINER[a]	PRESERVATION	HOLDING TIME	SAMPLE VOL, mL(minimum)[b]
pH	P,G	None	Measure in Field; 24 hours	50 (25)
Organolead	G-Amber with Teflon-lined	Cool, 4°C	14 days	1000, fully filled with zero headspace
Ortho-Phospate	P,G	Cool, 4°C	48 hours	50 (50)
Phosphate, Total	P,G	Cool, 4°C H_2SO_4 to pH <2	28 days	50 (50)
Silica	P,G	Cool, 4°C	28 days	50 (50)
Solids, Total Dissolved	P,G	Cool, 4°C	7 days	100 (50)
Sulfate	P, G	Cool, 4°C	28 days	50 (50)
Sulfide (Total)	P, G	Cool,4° C, 4 drops or more/100 ml of 2 N Zn acetate, 6 N NaOH to pH >9	7days	1000 (500) fully filled with zero headspace
Sulfide (Reactive)	P,G	May adjust to pH 12 w/ NaOH and add Zinc acetate but integrity of sample maybe affected, cool, store in dark.	Analyze as soon as possible.	100 (100), fully filled w/ zero headspace.
ORGANICS				
Semivolatile Organics (8270) (no residual chlorine present) Semivolatile	G	Cool, 4°C	7 days to extract; 40 days to anal. after extraction.	1-gal. or 2-1/2 gal. amber glass with Teflon liner
Semivolatile Organics (8270) (residual chlorine present)	G-Amber	Add 3ml of 10% sodium thiosulfate per gallon; Cool, 4°C	7 days to extract; 40 days to anal. after extraction.	1-gal. or 2-1/2 gal. amber glass with Teflon liner
Purgeable Organics (8260) (no residual chlorine present)	G(40 ml VOA vial with Teflon-lined septum cap	Adjust to pH<2 (see footnote c); Cool, 4°C	28 days	2- 40 ml VOA vials fully filled with zero headspace

Table 3.7-3 Sampling and Preservation for Water and Wastewater (Continued).

PARAMETER	CONTAINER[a]	PRESERVATION	HOLDING TIME	SAMPLE VOL, mL(minimum)[b]
Purgeable Organics (8260) (residual chlorine present)	G(40 ml VOA vial with Teflon-lined septum cap)	Cool, 4°C, adjust to pH<2, see footnotes c & d	14 days	2- 40 ml VOA vials fully filled with zero headspace
Purgeable Aromatics	G-V	Cool, 4°C, Adjust to pH<2, see footnotes c & d.	14 days	2 x 40 (40) vials fully filled with zero headspace
Purgeable Halocarbons	G-V	Cool, 4°C, see footnote d	28 days	2 x 40 (40) vials fully filled with zero headspace
Acrolein & Acrylonitrile	G-V	Cool, 4°C, pH 4-5, see footnote d	14 days	2 x 40 (40) vials fully filled with zero headspace
Gasoline/Diesel/TPH	G-V	Cool, 4°C HCl to pH<2, see footnotes c & d	28 days	2 x 40 (40) vials fully filled with zero headspace
N-Methyl-carbamate pesticides	G	Cool, 4^0 C, pH 4-5 with 0.1N chloro-acetic acid	7 days to extract; 40 days to anal. after extract.	1000 (1000)
Pesticides	G	Cool, 4°C,	7 days to extract; 40 days to anal. after extraction.	1000 (1000)
PCB	G	Cool, 4°C	7 days to extract; 40 days to anal. after extraction.	1000 (1000)
PCDD/PCDF	G	Cool, 4°C, see footnote d	7 days to extract; 40 days to anal. after extraction.	1000 (1000)
PAH (polynuclear aromatic hydrocarbons)	G	Cool, 4°C, store in dark, see footnote d	7 days to extract; 40 days to anal. after extraction.	1000 (1000)
Formaldehyde	G	Cool, 4°C,	7 days to extract; 40 days to anal. after extract.	500 (1000)
Chlorinated Phenols	G	Cool, 4°C, see footnote d	7 days to extract; 40 days to anal. after extraction.	1000 (1000)

Table 3.7-3 Sampling and Preservation for Water and Wastewater (Continued).

PARAMETER	CONTAINER[a]	PRESERVATION	HOLDING TIME	SAMPLE VOL, mL(minimum)[b]
Nitroaromatics	G	Cool, 4°C, see footnote d. Store in the dark.	7 days to extract; 40 days to anal. after extraction.	1000 (1000)
Oil & Grease	G	Cool,4°C, adjust to pH<2 w/ HCL, H_2SO_4 or $NaHSO_4$	28 days	1000 (1000)
Radioactivity Alpha, Beta and radium		HNO_3 to pH <2	6 months	1 gallon
TOX (Total Organic Halides)	G-Amber	Cool, 4°C, H_2SO_4 to pH<2, see footnote d.	28 days	2 x 250 (2 x 100) fill with zero headspace
TOC	P,G	Cool, 4°C, adjust to pH<2 with HCL, H_2SO_4 or $NaHSO_4$	28 days	100 (25)

[a] P = Polyethylene container with polypropylene closure.
G = Glass container with Teflon-lined closure.
G-V = Glass VOA vial or bottle with Teflon septum.

[b] Desired volume. Minimum volume for analysis shown in ( ).

[c] Acidification inhibits microbial degradation of aromatic compounds such as benzene, toluene, and ethyl benzene. However, hydrochloric acid irreversibly degrades the purge-and-trap system. If aromatic compounds are anticipated at low levels and the sample is not expected to be analyzed immediately, acidify sample with 8 drops 6 N HCL or 6N H_2S0_4 per 40 mL VOA vial, or add 0.25 g of $NaHSO_4$ per 40 mL VOA vial.

[d] If residual chlorine is present, preserve with $Na_2S_2O_3$. Use 1 drop of 10% w/v $Na_2S_2O_3$ per 40 mL VOA vial or 0.8 mL of 10% w/v $Na_2S_2O_3$ per liter of water.

3.8 SAMPLE SHIPMENT.

3.8.1 Transportation.

The sample transportation options available to collectors are:

1) Collector delivery to the designated laboratory.

2) DTSC has a contract with Federal Express/ to deliver environmental and hazardous samples to the specified laboratory. Contact Ramona Pam of ECL for more information at (510) 540-3580.

3.8.2 Packaging.

Samples are packaged and labeled for two broad classes of samples:

1) Environmental Samples.
2) Hazardous Samples.

Water samples, background soil samples and air samples are usually considered to be environmental samples. Many soil and solid samples may also be environmental samples, depending on the site. As a general rule, samples are considered hazardous unless known to be otherwise. All samples from drums, tanks, and process streams are considered hazardous unless known to be non-hazardous.

Environmental samples have minimal special packaging and marking requirements. More information is given in Appendix C.

Packaging, labeling and marking requirements for shipping hazardous materials are much more extensive. Packages must comply with the Code of Federal Regulations, Title 49, Parts 171 through 179. Most samples will be classified as "limited quantities" under part 173.118 or part 173.153. Some shipments with very small samples may qualify "small quantities" in section 173.4. Specific instructions for use of these regulations are contained in Attachment C of this manual.

The 49 CFR regulations apply to surface and air shipments, although regulations for air shipments are more restrictive. All individual air carriers have additional requirements including the use of International Air Transport Association Dangerous Goods Regulations. For all practical purposes, these regulations have superseded 49 CFR for air shipments. Additional information can be obtained from individual air carriers.

ECL maintains a Federal Express account that can be used to ship hazardous samples. If DTSC field offices anticipate shipping samples by air, at least one person in each office should be familiar with shipping regulations.

3.9 DOCUMENTATION.

Field notes including boring logs and actual sampling procedures should be completely documented in accordance with SW-846 requirements. Chain of custody should be consistent with ECL Chain of Custody procedure and SW-846. These documents should be properly reviewed and compiled. This validation process should be documented with the appropriate signatures.

3.9.1 Chain of Custody.

Sample chain of custody (COC) refers to all records maintained in the field and laboratory for sample identification, transmittal and receipt. When a sample is maintained under chain of custody, the possession of the sample can be traced from collection until disposal. This procedure is necessary to insure that the sample or data derived from the sample is admissible as evidence in legal proceedings.

A sample is considered under custody if:

1) It is in your possession, or
2) It is in your view after being in your possession, or
3) It was in your possession and you locked it up, or
4) It is in a designated secure area.

In order to establish that a sample is valid, it is also necessary to document the measures taken to prevent or detect tampering or loss of sample. Measures must also be taken to detect and prevent tampering and contamination to sampling equipment and the sample site. This is done by the use of evidence tape, locks, custody seals and documented observations.

Since it is not always possible to know in advance if a sample will be used as evidence, all samples are maintained under chain of custody. Use of standard operating procedures throughout the sampling process will contribute to the consistency and quality of the data produced.

3.9.2 Sample Identification.

Preprinted sample collection labels similar to Figure 3.9-1 are recommended to identify samples collected for shipment to ECL. Labels for all collected samples including replicates, field blanks and spikes, should be filled out completely. The minimum information should be:

1) Site name & location
2) Field ID no.
3) Collection date and time
4) Collector name
5) Preservation

3.9.3 Custody Seals.

Custody seals are strips of printed tape that are used to demonstrate that no tampering has occurred. Seals can be placed over container caps, bags containing samples, or sample transport containers. They may also be used to seal sampling equipment or the site (e.g. house doors).

3.9.4 Chain of Custody Forms.

There are many COC transfers during the course of a sampling program. In order to document these transfers, all samples should be accompanied by the Sample Analysis Request (SAR) form (Fig. 4.0-3). A chain of custody section is included in the form. The original form always travels with the samples and the initiator keeps a copy. In some instances, such as the collection of air samples on solid sorbents, it is necessary to establish chain of custody procedures before samples are collected.

The custody records are used for a packaged lot of samples. More than one form may be used if the number of samples or the number of transfers exceeds the capacity of the form. The purpose is to document the transfer of a group of samples traveling together. When the packaged lot is broken down or regrouped, a new chain of custody form must be added.

To use COC forms, the following procedure should be used:

1) The originator fills in all requested information.

2) The person taking custody checks the sample label information against the custody records, and the condition of sample container and seals. Any discrepancies should be noted and reported

3) The originator signs on line 1 of the COC section and keeps the triplicate (pink) copy.

4) The person taking custody signs on line 2 and each person receiving custody

thereafter signs on lines 3, 4, 5, etc.

5) In all cases, inclusive dates should be clearly shown for each custody transfer.

6) The original (white) and duplicate (yellow) COC copies (SAR) are kept with the samples.

Samples should be delivered to the laboratory as soon as practicable. This is usually within 1 or 2 days of collection but may be sooner depending on the analyses required. The samples are relinquished to the Lab sample custodian, who will verify the COC information and take custody. A unique laboratory number will then be assigned to each sample as it is log to a permanent log book.

If a discrepancy appears between sample labels and the chain of custody records, the person receiving custody should attempt to resolve the problem by checking all available information and then document the situation on the custody form and in the project notebook. Changes should be noted in the remarks section and should be initialed and dated.

Transfers of sample(s), extracts or digestates should be documented also. The sample I. D., person's name, and inclusive dates should be clearly shown.

Sample Label

Collector: _____

Sample No.: _____

Place of Collection: _____

_____

Date Sampled: _____

Time Sampled: _____

Field Information: _____

_____

Lab # _____

Preservation: _____

_____

_____

Figure 3.9.1

4.0. LABORATORY SERVICES

This section describes laboratory services available through Environmental Chemistry Lab (ECL). The Department laboratories include the Environmental Chemistry Lab (ECL), and ECL-Southern California (ECL-SC). ECL is the primary lab serving northern and central California, and ECL-SC is the primary lab serving southern California.

Services of a commercial laboratory are also available as described on the contract laboratory section.

The following four pages list individuals to contact for more information in each of the categories listed.

Environmental Chemistry Laboratory
California Department of Toxic Substances Control

Bruce La Belle, Ph.D., Chief
Thomas Li, Ph.D., Asst. Chief

Main Office:

700 Heinz Ave; Suite 100
Berkeley, CA 94710
Phone: (510) 540-3003

Telecopier: (510) 540-2305

Sample Receiving:

700 Heinz St., Suite 150
Berkeley, CA 94710
(510) 540-3610

(510) 540-3615

Environmental Chemistry Laboratory - Southern California (ECL-SC):

1449 W. Temple Street, Room 101
Los Angeles, CA 90026

Contact: Russ Chin
Phone: (213) 923-4879

Telecopier: (213) 580-5706

Information Contacts (alphabetical order)

Bioassay and Fish Bioassay:

Lorna Garcia	2441	
James Cheng	2337	

Biomonitoring:

Kim Hooper	3499	Cell: (510) 812-6546

Contract Laboratories:

Lorna Garcia	2441

Sample Transport Contacts:

Ramona Pam	3580

Data Interpretation, Data Validation, SAP and QAPP Review:

Lorna Garcia	2441
James Cheng	2337

Laboratory Information Management System (LIMS):

Jiong Cao	2925
Cuiyan Gan	3284

GC and LC Analyses:

Jarnail Garcha	3468	Cell: (510) 812-6554
Russ Chin (ECL-SC)	(213) 923-4879	

GC/MS Analyses:

William Lum	3060	Cell: (510) 812-6552
Russ Chin (ECL-SC)	(213) 923-4879	

Health and Safety:

Gurmail Sivia	3622
Martin Snider	5258, 2773

ICP and ICP/MS:

Jarnail Garcha	3468	Cell: (510) 812-6554

Immunoassays

Ruth Chang	3447	Cell: (510) 812-6558
Cindy Dingman	2329	

Inorganic Analyses - Metals, Waste Extraction Test, and Anions:

Jarnail Garcha	3468	Cell: (510) 812-6554

Lab Waste Management:

Gurmail Sivia	3622	
Thomas Li	2047	Cell: (510) 812-6260

Methods Development:

Jarnail Garcha	3468	Cell: (510) 812-6554
William Lum	3060	Cell: (510) 812-6552
Russ Chin	(213) 923-4879	

Mobile Laboratory:

Ruth Chang	2651	Cell: (510) 812-6558

Operations Management and Lead Programs' Liaison :

Martin Snider	5258

PCDDs and PCDFs Analyses:

Reber Brown	3322
Joginder Dhaliwal	(510) 849-5256

Quality Assurance / Quality Control:

Cindy Dingman	2329

Sample Management Officer(SMO):

Gurmail Sivia	3622
Martin Snider	5258

Sampling and Monitoring:

Myrto Petreas	3624

Special Studies:

Martin Snider	(510) 849-5258	
Myrto Petreas	3624	
Kim Hooper	3499	Cell: (510) 812-6546

Support Services and Secretarial Staff:

Linda Johnson	3003
Virginia Washington	3003

ECL User's Manual:

Cindy Dingman 2329

RELATED LABORATORY SERVICES

DHS Environmental Laboratory Accreditation Program (ELAP):

850 Marina Bay Parkway, Bldg P, 1st Floor
Richmond, CA 94804
Phone: (510) 620-3155
Fax: (510) 620-3165
George Kulasingam, Ph.D., Program Chief

Environmental Health Laboratory Branch:

California Department of Health Services
850 Marina Bay Parkway, Suite 6365 / EHL
Richmond, CA 94804
Phone: (510) 620-2800
Fax: (510) 620-2825

Primary Contacts for Air Sampling and Analysis:

Stephen Wall 3123
Diamon Pon 2639

Contract Laboratory:

Theresa Allen
Sequoia Analytical Laboratory
885 Jarvis Drive
Morgan Hill, CA 95037 (408) 776-9600

Diane Galvin
Advanced Technology Laboratory
3275 Walnut Ave.
Signal Hill, CA 90807 (562) 989-4045

4.1 ENVIRONMENTAL CHEMISTRY LABORATORY ANALYTICAL SERVICES.

4.1.1 Introduction.

ECL is a full service analytical chemistry laboratory determining metals, anions, pH, flash point, volatile and semivolatile organic compounds (EPA base/neutral, acid extractable organics) including petroleum products, pesticides, carbamates, solvents, and explosives. ECL also has expertise in the non-routine, highly specialized analysis of unknown hazardous waste, and in providing technical review and technical consultation to DTSC. New analytical methods are developed or implemented by ECL to anticipate site and waste characterization needs and to address new regulations and laws for hazardous waste management. Past examples include the development and implementation of analytical methods for percholorate, total organic halogens in oil, halogenated aromatic sulfonic acids by high pressure liquid chromatography, gasoline range organics and diesel range organics in contaminated soil, metals by ICP/MS, and work on EPA's TCLP extraction technique. ECL also evaluates new processes, working with the Pollution Prevention Program, for the treatment and recycling of hazardous waste. In addition, ECL provides basic and advanced training courses encompassing sampling plans and techniques, analytical procedures, and data interpretation.

4.1.2 Analytical Requests.

One of the major questions facing DTSC staff in their site investigations is to determine which analyses are required to properly characterize the samples they have collected. Frequently, the "shotgun" approach is used and comprehensive analysis is requested. These may include volatile organics analysis (VOA), acid, base/neutral extractables, chlorinated and organophosphorus pesticides, carbamates, herbicides, polynuclear aromatic hydrocarbons (PAH), nitrophenols, trace metals and anions. Although the analyses mentioned above are routine, they do involve nine different analytical procedures, seven different sample preparations, and seven different instruments. Total analytical time, hence turnaround time, will be lengthy, making it difficult to assign a high priority level to the request.

A more efficient approach would be to request analysis for a specific class or classes of compounds based on site history, field observation and/or field screening results. Information about the site may be obtained from the types of wastes stored or disposed at the site. From the industrial or manufacturing processes, information can usually be derived about the composition of the wastes generated. Hazardous waste manifests, business records, and container labels also provide important information about waste composition. Another valuable reference is the Kirk-Othmer "Encyclopedia of Chemical Technology".

Field observations and field screening methods are particularly useful in determining which laboratory analysis to request. Field screening and hazardous characterization should be performed, as much as possible, on site when gathering information about the samples. Organic versus inorganic classifications may be determined using field testing procedures. When screening of volatile organics, field screening instruments, *e.g.* OVA, HNU, Miran, etc., should be used, if possible. Analytical requests should be made based on the information obtained. As an example, an analysis request for solvents and/or semivolatile organics is advisable if a sample matrix is found to be organic by initial screening tests. Metal analysis should not be requested on this sample unless organometallic compounds are suspected to be present.

Requestors can also rely on the expertise of ECL's analytical unit supervisors, both in Berkeley and Los Angeles to determine the type and level of analysis required. Consultation with ECL or ECL-SC is highly recommended before sampling.

Since July 1, 1990, the annual allocations of analytical lab services budgeted for DTSC have been based on the concept of the Laboratory Cost Unit (LCU). There are two types of LCUs charged to any procedure requested: the Basic Charge and the Analytical Charge. The first charge reflects instrumental and other preparatory work, and the second charge is reflective of the analyst's time and relative complexity of the procedure. Figures 4.0-1 and 4.0-2 list the most frequently requested analytical procedures and the corresponding Basic and Analytical charges in LCUs for each batch of ten samples or a fraction thereof. The total number of LCUs charged for a given procedure also includes necessary QC work.

Analytical requests should be made in accordance with the protocol specified in the "ECL Express Plan" (EEP) which is detailed in Appendix A. The purpose of the protocol is to match the analytical request (workload) with a lab (State lab or a contract lab), that has the capacity to perform the analysis within the required time frame. In the event that the lab capacity is not available, options will be thoroughly discussed with the requestor. An authorization for sample analysis must be obtained by the requestor before laboratory analysis begins.

To obtain an authorization for sample analysis, the requestor must submit, by FAX at (510) 849-5271 or e-mail, an Authorization Request Form (ARF) to ECL's Sample Management Officer (SMO), e-mail to GW's "Sample Mgmnt" mail station. Within two working days of receiving the ARF, the SMO will locate a laboratory available to perform the analysis and issue an Authorization Number (AN) for the request. It is essential that the requestor indicate the objective(s) of the analysis and specify what quantitation limits are needed on the ARF, Part A (Attachment 2, Appendix A) and on the SAR, line 14 (Fig.4.0-2). The information provided will help the laboratory to determine which sample preparation and analytical method to use. Requesting an analysis procedure that is additional to the original authorization will require a re-authorization by the SMO before the laboratory proceeds with the analysis. The ARF and SAR forms are available

on LAN's "T" drive under T:Forms/ECL/ARForSAR form.

Quantitation Limits for routine analytical procedures are provided in Appendix B. These are only approximations and are highly dependent on the nature of the matrix. If a lower quantitation limit is needed be sure to call ECL (the contacts and phone numbers are listed in Section 4.0-Laboratory Services. The Sample Analysis Request (SAR) form (Rev. 6/00, which must accompany all samples submitted to the laboratory, is used to provide some information on the samples collected, specifies the analysis required, and documents the chain of custody (see Figure 4.0-2). Instructions, code designations, SMO and lab telephone numbers and container information are printed on the reverse side of the SAR form. To prevent potential delays on laboratory sample analysis, it is also essential that the requestor enter all applicable codes and information, including analysis objectives and special detection limit requirements on the SAR.

For information on sampling, sample containers, sample preservation, documentation and sample shipment, refer to Section 3. Samples should be collected and shipped directly to the designated laboratory no later than five working days past the expected delivery date indicated on the ARF. Please note that samples with short holding times (consult with Tables 3.7-2 and 3.7-3) should be delivered **as soon as possible** after collection to the designated laboratory, or the laboratory may not be able to meet the sample holding time requirements.

Samples are routinely saved by the laboratory until permission is received from the requestor authorizing the proper disposal of the samples. A Sample Disposal Form is sent to the requestor by the laboratory soon after the final analysis is completed. The requestor should complete the form and return it to the laboratory. Sample Storage capacities (State and commercial laboratories) are limited, **so requestors are advised to promptly inform the laboratory of the sample disposition**. Samples that may be used as evidence in legal proceedings should be saved until the suit is settled.

Table 4.1-21 at the end of this section contains the EPA, ECL, ECL-SC, and contract laboratory equivalent method numbers for organic, inorganic, and miscellaneous analytical methods. Table 4.1-22 contains the EPA equivalent analytical method numbers for solid waste, wastewater, and drinking water. The status of ECL In-House Methods is listed in Table 4.1-23. The status of ECL SOPs is listed in Table 4.1-24.

Figure 4.0-1

ENVIRONMENTAL CHEMISTRY LABORATORY
ANALYTICAL PROCEDURES AND CORRESPONDING
LAB COST UNITS (L C Us)

PROCEDURE	CODE	CHARGES	
		BASIC L C Us	ANALYTICAL LCU / SPL
Anions Scan(F,Br,Cl,NO3,NO2,SO4,PO4)	C	7	2
Carbamates	E	29	6
Cations Scan	F	7	2
Chlorophenols (TCP, PCP)	G	18	6
Cyanides	H	10	4
Diesel, GC/FID	I	16	7
Dinitrocompounds, HPLC	J	21	7
Dioxins and Furans	K	35	15
EDB and DBCP	L	15	5
Extractable Organics (BNA)	N	40	7
Gasoline, GC/PID/FID	P	16	7
Herbicides (Chlorinated)	Q	30	15
Ignitability	R	3	2
Metals Scan	S	12	2
Metals, Specific (1-2 Metals)	T	10	2
Moisture, %	U	2	1
Non-Target Organics, LC/MS	V	Variable	Variable
Oil and Grease	W	7	4
Organolead (FAAS)	X	10	2
Organochlorinated Pesticides	Y	24	10
Organophosphorus Pesticides	Z	24	10

prolcuwk.3nly.10/95

Figure 4.0-1 con't

ENVIRONMENTAL CHEMISTRY LABORATORY
ANALYTICAL PROCEDURES AND CORRESPONDING
LAB COST UNITS (L C Us)

PROCEDURE	CODE	CHARGES	
		BASIC L C Us	ANALYTICAL LCU / SPL
P A Hs	ZA	36	11
P C Bs	ZB	21	8
pH	ZC	2	1
Semivolatile Organics, GC/MS	ZE	45	12
Specific Conductance	ZF	2	1
TCLP	ZG	Variable	Variable
Total Petroleum Hydrocarbon (TPH)	ZH	8	3
Total Organic Halides (TOX)	ZI	7	5
Volatile Organics, GC/MS (Cap.Column)	ZK	15	5
Waste Extraction Test (W.E.T),California	ZO	12	3
Misc. Procedures such as: Particle Sizing, reactivity, Ethylene Glycol Confirmation of Non-target Compounds, etc.	ZZ	Variable	Variable

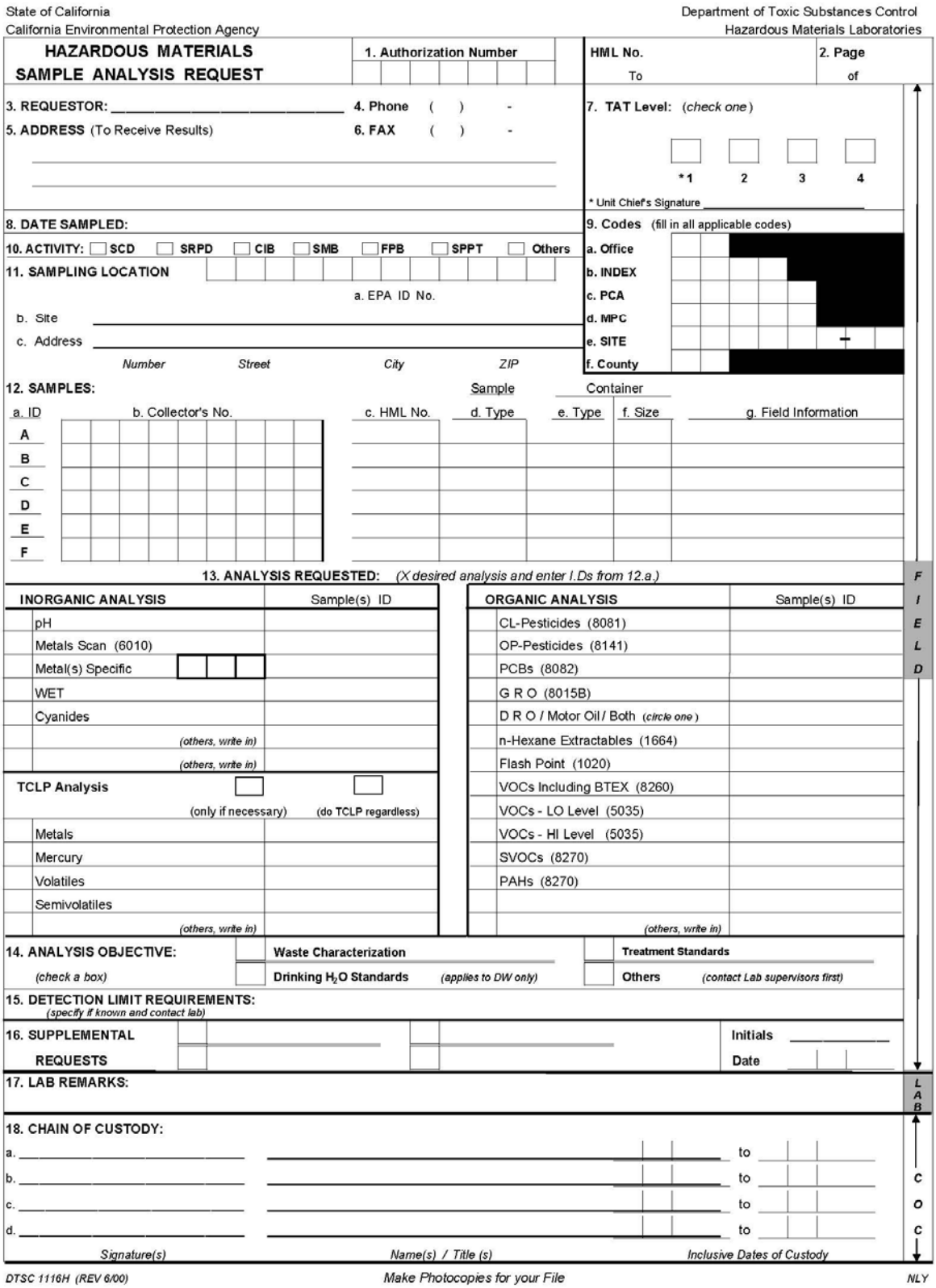
prolcuwk3.nly.10/95

BASIC CHARGE:
Reflects the expenditure of lab resources for instrument set-up, calibration and QC work for each batch of 10 samples (4 for Dioxins).

ANALYTICAL CHARGE:
Based on actual measurement of expenditure of lab resources for performing the sample analysis.

TOTAL CHARGE FOR A BATCH OF SAMPLES, LCUs =

Basic Charge **x** n/10 or 4 **+** Analytical Charge **x** n
Where n = number of samples



State of California
California Environmental Protection Agency

Department of Toxic Substances Control
Hazardous Materials Laboratories

HAZARDOUS MATERIALS SAMPLE ANALYSIS REQUEST

1. Authorization Number

HML No. / To

2. Page / of

3. REQUESTOR: _____ 4. Phone ( ) -

5. ADDRESS (To Receive Results) 6. FAX ( ) -

7. TAT Level: (check one)

*1 2 3 4

* Unit Chief's Signature _____

8. DATE SAMPLED:

9. Codes (fill in all applicable codes)

10. ACTIVITY: SCD SRPD CIB SMB FPB SPPT Others

11. SAMPLING LOCATION

a. EPA ID No.

b. Site

c. Address

Number Street City ZIP

a. Office
b. INDEX
c. PCA
d. MPC
e. SITE
f. County

12. SAMPLES:

a. ID	b. Collector's No.	c. HML No.	Sample d. Type	Container e. Type	f. Size	g. Field Information
A						
B						
C						
D						
E						
F						

13. ANALYSIS REQUESTED: (X desired analysis and enter I.Ds from 12.a.)

FIELD

INORGANIC ANALYSIS	Sample(s) ID
pH	
Metals Scan (6010)	
Metal(s) Specific	
WET	
Cyanides	
(others, write in)	
(others, write in)	

TCLP Analysis (only if necessary) (do TCLP regardless)

Metals	
Mercury	
Volatiles	
Semivolatiles	
(others, write in)	

ORGANIC ANALYSIS	Sample(s) ID
CL-Pesticides (8081)	
OP-Pesticides (8141)	
PCBs (8082)	
G R O (8015B)	
D R O / Motor Oil / Both (circle one)	
n-Hexane Extractables (1664)	
Flash Point (1020)	
VOCs Including BTEX (8260)	
VOCs - LO Level (5035)	
VOCs - HI Level (5035)	
SVOCs (8270)	
PAHs (8270)	
(others, write in)	

14. ANALYSIS OBJECTIVE: (check a box)
Waste Characterization; Drinking H_2O Standards (applies to DW only); Treatment Standards; Others (contact Lab supervisors first)

15. DETECTION LIMIT REQUIREMENTS: (specify if known and contact lab)

16. SUPPLEMENTAL REQUESTS

Initials _____ Date

17. LAB REMARKS:

LAB

18. CHAIN OF CUSTODY:

	Signature(s)	Name(s) / Title (s)	Inclusive Dates of Custody
a.			to
b.			to
c.			to
d.			to

COC

DTSC 1116H (REV 6/00) Make Photocopies for your File NLY

Figure 4.0-2 Con't

INSTRUCTIONS

1. **Authorization No.** - Enter the number acquired from HML's STO.
2. **Page —of—** - Enter number of this page and the total number of pages to complete this request.
3. **Requestor's Name** - Print.
4. **Phone** - Enter Area Code and Phone Number of the Requestor.
5. **Address** - Where results should be sent.
6. **FAX** - Number to FAX results to.
7. **TAT Level** - check one : Turnaround Time Level.
   Level 1: 15 days and requires the signature of Unit Chief on SAR.
   Level 2: 30 days, Level 3: 45 days and Level 4: when possible.
8. **Date Sampled** - Enter date of sample(s) collection.
9. **Codes** - All applicable codes must be entered
   a. See Table II below.
   b-e . See DTSC lists of these codes.
   f. Enter County Code from Table I below.
10. **Activity** - Check the appropriate box to indicate DTSC activity generating sample(s). Check "Others" for unlisted and Non-DTSC samples.
11. **Sampling Location** - Where sample(s) are collected
    a. EPA ID No. - Enter U.S.EPA twelve-digit identification no. for the site
    b. Site - Enter nameof generator, facility or site.
    c. Address - Enter address of generator, facility or site designated in 11 b. Enter two-digit County Code in item 9 f.
12. **Samples** - Enter specific information as indicated.
    a. ID - Predesignated Line Identifier.
    b. Collector's No. - Enter the collector's sample number(s). Number should NOT exceed 9 characters.
    c. Lab No. - For Lab use only.
    d. Type - Enter sample type, e.g., sludge, soil, etc.
    e. Container Type - Enter appropriate container codes from Table III
    f. Container Size - Enter appropriate container size from Table III. Full size is required for analysis.
    g. Field Information - Enter information significant to personnel safety and analysis requested, e.g., cyanide contamination suspected, air volume if applicable, etc.
13. **Analysis Requested** - Check one or more of the boxes as applicable. For each box checked, enter the line identification code(s) (item 12 a.) to designate the sample(s) to be analyzed, e.g.,
    ☐ pH A,C,H.
14. **Analysis Objective** - Check one.
15. **DL Requirements** - Specify, and contact Lab if unusual.
16. **Supplemental Requests** - Enter procedures additionally requested, line or sample ID, initial and Date for Supplemental Requests ONLY.
17. **Lab Remarks** - For Lab use only.
18. **Chain of Custody (COC)** - Chronologically, the person(s) who had custody of the sample(s) should enter information above the lines as designated. Item 18 c. is for the latest in the chain of custody.

Table I - CALIFORNIA COUNTY CODE NUMBERS (Item 9f)

Code	County	Code	County	Code	County
01	Alameda	20	Madera	39	san Joaquin
02	Alpine	21	Marin	40	San Luis Obispo
03	Amador	22	Mariposa	41	San Mateo
04	Butte	23	Mendocino	42	Santa Barbara
05	Calaveras	24	Merced	43	Santa Cruz
06	Colusa	25	Modoc	45	Shasta
07	Contra Costa	26	Mono	46	Sierra
08	Del Norte	27	Monterey	47	Siskiyou
09	El Dorado	28	Napa	48	Solano
10	Fresno	29	Nevada	49	Sonoma
11	Glenn	30	Orange	50	Stanislaus
12	Humboldt	31	Placer	51	Sutter
13	Imperial	32	Plumas	52	Tehama
14	Inyo	33	Riverside	53	Trinity
15	Kern	34	Sacramento	54	Tulare
16	Kings	35	San Benito	56	Ventura
17	Lake	36	San Bernardino	57	Yolo
18	Lassen	37	San Diego	58	Yuba
19	Los Angeles	38	San Francisco		

Table II
DTSC Offices
(Item 9 a)

Sacramento	01
Fresno	1F
Berkeley	02
Glendale	03
Cypress	04
HQ Units	05

HML
Phone & FAX Numbers

HML Berk (510) 540-3003

HML L.A. (213)580-5796

HML STO (510)540-3111
FAX (510)849-5271

Table III - Container Type and Size (Items 12 e and 12 f)

	Liquid Samples		Solid Samples	
	Type	Size	Type	Size
Organic Analysis, Gen	G	1000 ml	G	250 mg
Organic Analysis, VOA	G-V	40 ml	G-V	40 gm
Organic Analysis, Tox	Ambr G-V	100 ml	Ambr G-V	40 gm
Inorganic Analysis	P, G	1000 ml	G	250 gm

P=Polyethyene Container & Closure G=Glass Container with Teflon Closure G-V=Glass VOA vial or bottle with Teflon Septum

,DTSC 1116H (REV 6/00) *Instructions*

Figure 4.0-2 Con't

4.1.3 ECL Capabilities.

ECL can routinely analyze samples for the 17 elements and the volatile and semivolatile organics listed in Tables I, II and III of Title 22, California Code of Regulations (5/31/91), 66261.24(a)(1)(B), (2)(A) and (2)(B). ECL also excels in performing non-routine analysis, such as in characterizing and identifying unknown wastes. An example of past work was the analyses of unknown waste samples taken from drums Stored at Pier 70, San Francisco. Screening tests were initially employed to separate the samples into organic or inorganic classifications. Further analyses identified polyamines, organic acids, sodium sulfite and polychlorinated biphenyls (PCBs) in some of the samples. These results were used in the successful criminal conviction of the responsible party.

The following categories of analysis are routinely performed in ECL. Classification and identification of non-routine hazardous materials are also performed in this laboratory by special requests.

4.1.3.1 Physical Testing.

1) Acidity/Alkalinity: This test is used to measure the amount of strong acid or base in a sample. Results are reported in units of milliequivalents/gram or millequivalents/liter.

2) Asbestos: By special arrangement, asbestos in a sample can be analyzed by Contract Laboratories. Samples should be submitted to the Contract Laboratory.

3) % Dry Solids: ECL Method 704-S may be used to determine the weight percent of dry solids in a soil, sediment or solid waste sample. Beginning October 1993, all soil samples received by the laboratory have been routinely analyzed for % dry solids. The result obtained in this procedure may be used to convert the wet-weight concentration[1] of an analyte (concentration obtained from the wet sample) to a dry-weight concentration as described in the following equation.

$$\text{Dry weight conc.} = \frac{\text{Wet weight conc.}}{\text{\% dry solids}} \times 100$$

[1] Note: Analyte concentration results are reported on a wet-weight basis unless requested otherwise.

4) Ignitability: EPA Method 1020 is used to characterize the flammability of liquids. This test is applicable to liquids only. Flash points less that ambient temperature are reported as, e.g., "<70^{o}F." Flash points greater than 140^{o}F (the hazardous waste criterion) are reported as ">140^{o}F."

   ECL Method 720 is also available to characterize the flammability of compressed gasses in aerosol products. The method is based on ASTM Standards D 3074-72 and D 3065-72.

5) Melting Point: This test is useful as a supplemental tool in the identification of pure solids.

6) pH: This test is used to determine if the sample is corrosive. For soil samples, our method involves measuring the pH of the aqueous solution or of a 1:1 mixture of soil and water, as specified in the Title 22 hazardous waste criteria.

7) Radioactivity: By special arrangement, radioactivity in a sample can be determined by the Sanitation and Radiation Laboratory (SRL) in Berkeley.

8) Reactivity: The test performed is dependent on the reactivity characteristic(s) of the sample. Tests may be performed to determine if a sample is dangerously reactive with water or generates toxic fumes when mixed with water. Tests for reactive cyanides and sulfides may also be performed.

9) Specific Conductance: This method is used to measure the specific conductance of drinking, ground, surface, and saline waters, domestic and industrial aqueous wastes and extracts or saturated paste extracts of soils.

4.1.3.2 Organic Analysis.

Prior to collecting samples for organic analysis, the collector must review all pertinent information such as field data, history of the sampling site, previous analytical data, and regulation requirements in order to determine the types or classes of organic compounds that are of interest and are likely to be present. Whenever possible, individual organic compounds, or a specific class of organic compounds, should be requested rather than the general characterization of a sample. The collector should also submit any relevant field/sample related information, as well as the objective of the sampling to the laboratory. This information is valuable to the laboratory staff in selecting the proper analytical method and instrument configuration that is optimized for the target analytes.

Analytical instruments available for organic analysis in ECL include gas chromatographs with a variety of detectors including mass spectrometers, liquid chromatographs, and an infrared spectrophotometer.

Organic methods in ECL are classified into two basic categories: volatile analysis and

semivolatile analysis.

1) VOLATILE ORGANIC ANALYSIS

Gasoline Range Organics (GROs):

Gasoline range, or purgeable organics in environmental samples are analyzed by purge-and-trap GC/FID technique (EPA 8015B).

GC-MS Scan for Volatiles:

This method is appropriate for the characterization and identification of unknown volatile organic compounds in hazardous waste samples. It is useful when prior history or information on chemical contamination of the sampling site is unavailable or unknown.

Although GC-MS scan uses the same analysis conditions as Method 8260, the method is qualitative. A tentative identification is assigned only if the electron impact mass spectrum of the unknown matches a spectrum from the mass spectral database of 43,000 organic compounds.

If no match occurs, the fragmentation pattern of the unknown is examined to determine if characterization is possible by identifying specific classes of organic compounds (*e.g.*, hydrocarbons, aliphatic, aromatic, etc.)

By special request and with ECL approval, non-target analytes can be confirmed and quantitated if the reference standards are available.

EPA Method 8260B - Volatile Organics by GC-MS (Table 4.1-1):

This is a GC-MS purge-and-trap quantitative method for volatile organics. The volatile components of a sample are purged with helium and concentrated in a sorbent trap. The trap is then rapidly heated to introduce the volatiles into the GC-MS system. The volatile mixture is separated in a capillary GC column and electron impact mass spectroscopy is used to identify the volatile compounds.

This method is used to quantitate volatile organic compounds (VOCs)that are insoluble or slightly soluble in water. Sample preparation procedures are dependent on the matrix types. An aqueous sample is purged directly, a soil sample is extracted with methanol and an aliquot of the methanol extract is purged. An organic liquid sample or a sample highly contaminated with organics must be highly diluted before analysis. Quantitation limits are therefore dependent on the sample matrix type and organic content.

It is important to note that GC/specific-detectors (*e.g.*, photoionization or electrolytic conductivity) usually provide lower detection limits than GC-MS.

These methods generally use GC-MS confirmation except at very low levels below GC-MS detection.

ECL Method 850 - Volatile Organics by Headspace GC-MS:

A septum vial containing the sample is heated to release the volatile organic components. An aliquot of the vial's headspace is injected into a GC-MS system. The GC column separates the volatiles into individual organic compounds. As each organic compound elutes from the column, it is subjected to electron impact ionization to produce a characteristic mass spectrum. The resulting mass spectrum is used to search against a mass spectral library for organic compound identification.

The method is useful for identification and characterization of volatile organics. Unknown volatiles in less contaminated samples can be individually identified and qualitatively confirmed, if reference standard materials are available. For highly contaminated samples where complete chromatographic separation is not possible, this method can yield important information as to which class or classes of volatile organics are present (i.e., chlorinated solvents, oxygenated solvents, aliphatic hydrocarbons or aromatic hydrocarbons). The proper analytical method can then be selected for optimal detection and quantitation.

3) **SEMIVOLATILE ORGANICS**

Diesel or diesel range organics (DROs) and motor oil or motor oil range organics (MOROs):

Diesel and motor oil range organic are analyzed by extraction of semivolatile hydrocarbons with methylene chloride and determination by GC/FID (ECL SOP 816-S). Tentative diesel and motor oil identification is based on chromatographic pattern recognition. Quantitation is done by comparing total peak area of the sample chromatogram with an external diesel or motor oil standard. This is a **compound non-specific, semi-quantitative analysis**.

EPA Method 1664: N-Hexane Extractable Material (HEM) and Silica Gel Treated N-Hexane Extractable Material (SGT-HEM) by Extraction and Gravimetry(Oil and Grease and Total Petroleum Hydrocarbons)

Concentrations of specific compounds or substances are not measured. Rather, groups of substances with similar characteristics are determined gravimetrically, based on their common solubility in *n*-hexane. In order to be measured, the substance must:

1. be soluble in *n*-hexane.
2. be extractable from an acidified sample.
3. not be volatilized (lost) during the sample work up. Applicability is limited

to semivolatile compounds.

GC-MS Scan for Semivolatiles:

This method is appropriate for the characterization and identification of unknown semivolatile organic compounds in hazardous waste samples. It is useful when prior history or information on chemical contamination of the sampling site is unavailable or unknown.

Although GC-MS scan uses the same analysis conditions as Method 8270, the method is qualitative. A tentative identification is assigned only if the electron impact mass spectrum of the unknown matches a spectrum from the mass spectral database of 43,000 organic compounds.

If no match occurs, the fragmentation pattern of the unknown is examined to determine if characterization is possible by identifying specific classes of organic compounds (*e.g.*, hydrocarbons, aliphatic, aromatic, etc.)

By special request and with ECL approval, non-target analytes can be confirmed and quantitated if the reference standards are available.

EPA Method 8270C - Semivolatile Organics by GC-MS (Table 4.1-2):

This method is used for the determination of semivolatile organics by GC-MS. The method involves sample extraction with methylene chloride, followed by GC-MS analysis for specific (target) compounds and for major non-target compounds (tentative identification). There are about 250 compounds listed in this method. ECL routinely analyzes samples for 67 compounds commonly found in hazardous waste streams. Others on the SW-846 list are by special request.

EPA Method 8082-Polychlorinated Biphenyls (PCBs) (Table 4.1-3):

Aroclor is the trade name for the PCBs manufactured by Monsanto. The first two digits of the four digits represent the types of molecules (12=chlorinated biphenyls); the last two digits represent the weight percent of chlorine (Aroclor 1221 = 20.5 to 21.5% of Cl, Aroclor 1260 = 60% of Cl). The only exception is Aroclor 1016 which does not follow the above description but rather contains 41% Cl by weight. The Aroclors listed in Table 4.1-4 are analyzed by GC/ECD. Confirmation for PCB is generally accomplished through pattern recognization of the eluting peaks. However, when matrix effects interfere with pattern recognition, confirmation using a second column or GC-MS may be necessary.

EPA Method 8081A-Chlorinated Pesticides and Toxaphene (Table 4.1-4):

Toxaphene and 20 chlorinated pesticides are routinely analyzed by capillary GC/ECD. Quantitation and confirmation are performed by a simultaneous two column analysis. (Toxaphene and congener-specific PCB analysis is available upon request. For information see special Analysis Requests section 4.1.3.4)

EPA Method 8141A - Organophosphorus Pesticides (Table 4.1-5):

Twenty-three organophosphorus pesticides are routinely analyzed by capillary GC using a flame photometric detector (FPD) operating in the phosphorus mode. Confirmation is done with a second column and a nitrogen/phosphorus specific detector (NPD).

EPA Method 8151A - Chlorinated Herbicides (Table 4.1-6):
Ten chlorinated herbicides are analyzed in this method. This very lengthy procedure involves extraction, hydrolysis and derivatization with diazomethane, followed by GC/ECD analysis. Confirmation is accomplished by a second column analysis or GC-MS if needed.

EPA Method 8011 - Ethylene Dibromide (EDB) and Dibromo-chloropropane (DBCP):

EDB (CAS # 106-93-4) and DBCP (CAS # 96-12-8) are extracted from soils and aqueous samples by hexane extraction. Analysis is by GC/ECD. Confirmation is performed by a second column analysis or by GC-MS.

EPA Method 8310 - Polynuclear Aromatic Hydrocarbons (PAH or PNA)(Table 4.1-7):

Sixteen PAHs are analyzed by HPLC using the UV and fluorescence detectors. Confirmation, if necessary, is performed by GC-MS. This analysis should only be requested if detection limits below those of Method 8270 are required. If PAH data are needed at low ppb quantitation limits (as in risk assessment), the requestor should note this requirement on the analysis request form so that the low level preparation procedure will be employed by the lab. These very low quantitation limits cannot be confirmed by GC-MS and will be confirmed by a second column technique.

EPA Method 8330-Nitroaromatics and Nitramines (Table 4.1-8):

Thirteen analytes are analyzed by HPLC using a UV detector. Confirmation is performed by a second column analysis.

ECL Method 734-N-Methylcarbamate Pesticides (Table 4.1-9):

Ten carbamate pesticides are analyzed by HPLC with post-column derivatization and fluorescence detection. Confirmation is normally not required due to the specificity of the method.

ECL Method 736-Dinitroaromatic Compounds and Dinoseb (Table 4.1-10):

Twelve dinitroaromatic compounds, including some selected herbicides and fungicides, are analyzed by high performance liquid chromatography (HPLC) with a UV detection. Confirmation is performed by thin-layer chromatography (TLC) or by GC-MS. ECL is also working on a HPLC/MS confirmation procedure.

ECL Method 740 - MOCA:

4,4'-methylenebis(2-chloroaniline) (MOCA), CAS # 101-14-4, in soils is analyzed by a HPLC/UV technique developed at ECL. Confirmation is by GC or GC-MS.

Guide for the Analysis of Petroleum Hydrocarbon Residues:

Use the table below as a guideline for requesting the analysis of petroleum hydrocarbon residues or oily residues.

GUIDE FOR THE ANALYSIS OF PETROLEUM HYDROCARBON RESIDUES

Suspected	Water	Solids Soil, Sludge	What is Measured ?	Is method constituent specific?	Is method quantitative?
Gasoline / GROs	EPA 8015B (GC/FID)	EPA 8015B	gasoline & GROs	No	semi**
Diesel / DROs	EPA 8015B (GC/FID)	EPA 8015B	diesel & DROs	No	semi**
Motor oil / MOROs	ECL 816-S (GC/FID)	ECL 816-S	Motor oil & MOROs	No	semi**
BTEX , Aromatics	EPA 8260B (GC/MS)	EPA 8260B	BTEX, aromatic VOCs	Yes	Yes
TPH	EPA 1664 (Gravimetric)	EPA 1664 (modified)	n-hexane extractable -silica gel treated non-volatile petroleum hydrocarbons	No	semi**
Oil and Grease	EPA 1664 (Gravimetric)	EPA 1664 (modified)	n-hexane extractable non-volatile organics (petroleum hydrocarbons, oils, animal fats, vegetable oils, waxes & related matter.)	No	semi**
PAHs	EPA 8270C (GC/MS) EPA 8310 (HPLC)	EPA 8270C EPA 8310	polynuclear aromatic hydrocarbons (8310 normally offers lower QLs than 8270C)	Yes Yes	Yes Yes
Unknown volatile organics	EPA 8260B (GC/MS)	EPA 8260B	volatile organics	Yes	Yes*
Unknown semivolatile organics	EPA 8270C (GC/MS)	EPA 8270C	semivolatile organics	Yes	Yes*

NOTES:

GROs = gasoline range organics, C6 - C12
DROs = diesel range organics, C10 - C23
MOROs = motor oil range organics, C19 - C32

VOCs = volatile organic compounds
TPH = total petroleum hydrocarbons
QLs = quanititation limits
* = semi-quantitative for non-target analytes
** = since constituents are non-specific, the results are not analyte specific

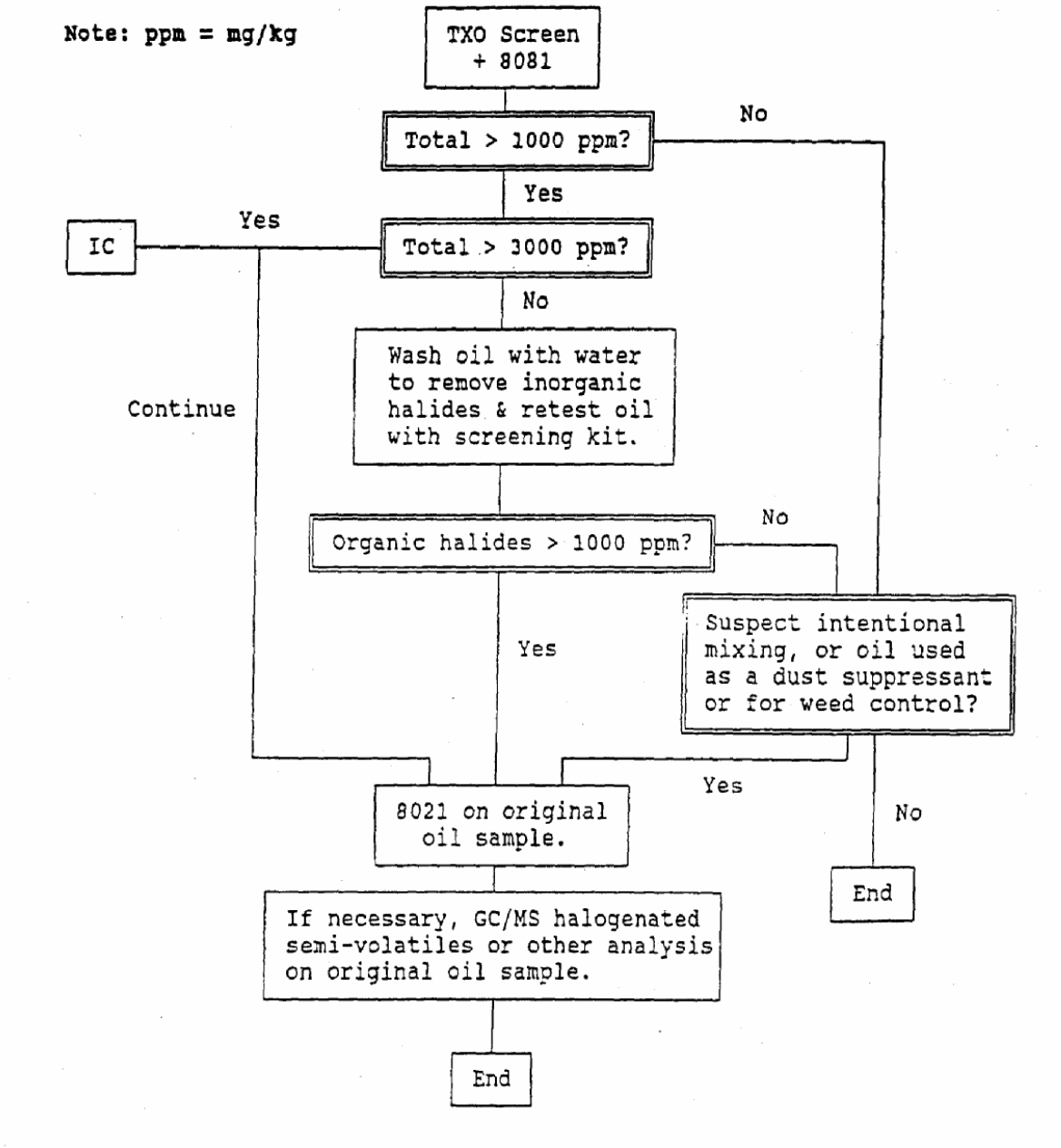
ECL Method 792 - Total Halogens in Oil (TXO):

The Chlor-D-Tect kit is used to screen oil samples for total halogen content. Results from this screening procedure are reported as ppm chloride, regardless of the actual halogen present (fluoride not included). The TXO procedure actually involves a number of sequentially assigned analytical procedures, depending on the outcome of the Chlor-D-Tect screening. Since PCBs are regulated at a much lower level than total organic halogens, a PCB analysis is automatically performed regardless of the TXO level. Further analysis is generally not required if the screening result indicates a halogen level ≤1000 ppm. On the other hand, a result >1000 ppm requires that additional steps be taken as illustrated in the following diagram.

EPA Method 8315 - Formaldehyde (carbonyl compounds):

Formaldehyde analyzed by HPLC and UV detection. Confirmation is not performed, usually samples are collected from known background contamination.

Total Halogens in Oil Analysis



Guide for Requesting the Toxicity Characteristic Leaching Procedure (TCLP) - GC, HPLC, and GC-MS methods:

The TCLP is an extraction procedure using an acetic acid buffer solution that is designed to test the leachability of solid waste for certain toxic organic and inorganic (see Section 4.1.3.3. Part 5) constituents. The TCLP extract produced is treated as a liquid sample and analyzed by the appropriate methods. Oily wastes warrant special consideration. Because oily wastes present a difficult problem in the TCLP (premature clogging of the filter during filtration), a "totals" analysis for the analytes of concern will be performed first to determine if the TCLP is required.

A request for TCLP organics should be specified by the compound class or the analyte(s) suspected to be in the sample. For this analysis, only the TCLP analytes will be analyzed and quantitated. Please use the following categories.

TCLP Volatiles:

TCLP extracts are screened by EPA Method 8260. Quantitation and confirmation are performed by EPA 8260 for only the TCLP volatiles listed in Table 4.1-11.

TCLP Semivolatile Acid/Base/Neutrals:

TCLP extracts are analyzed by EPA Method 8270 acid/ base/neutrals for only the TCLP semivolatile acid/base/neutrals listed in Table 4.1-12. If the Sample Analysis Request does not specify which TCLP-semivolatile group is to be determined, both TCLP semivolatile base-neutrals and acids are determined.

TCLP Cl Pesticides (GC):

TCLP extracts are analyzed by EPA Method 8081 for only the TCLP chlorinated pesticides listed in Table 4.1-13.

TCLP Herbicides (GC):

TCLP extracts are analyzed by EPA Method 8150 for only the TCLP herbicides listed in Table 4.1-14.

4.1.3.3 Inorganic Analysis.

1) Metals:

If significant levels of toxic metals are suspected, request "Metals, WET if necessary."

A total metals analysis is first performed to determine if a sample is hazardous by exceeding the corresponding Total Threshold Limit Concentration (TTLC) values for metals listed in Table II, Title 22, California Code of Regulations (5/31/91), 66261.24(a)(2)(A) . If the result is less than the TTLC, but greater than 10 times the corresponding Soluble Threshold Limit Concentration (STLC) value, a Waste Extraction Test (WET) will be performed (see 4.1.3.3. Part 4). If the concentration of a substance in the (WET) extract (in mg/L) is greater than the corresponding Soluble Threshold Limit Concentration, then the sample is "hazardous". The scheme for the metals analysis is shown below in Figure 4.4-1.

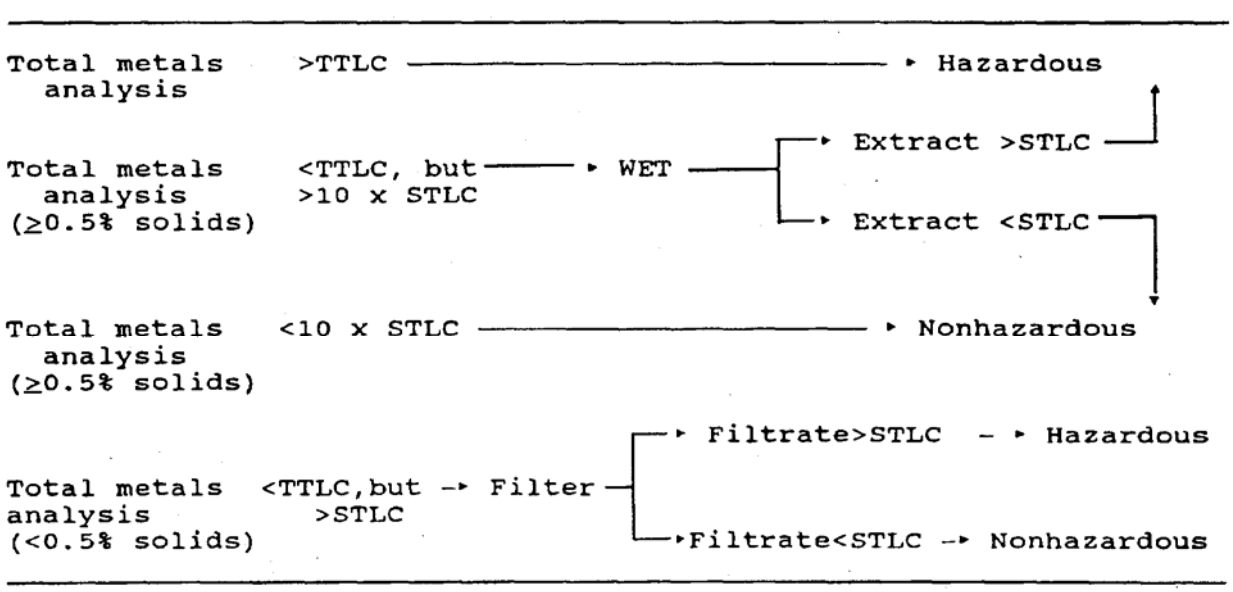


Figure 4.4-1. Metals Analysis Scheme

Table 4.1-15 lists the 14 metals that are routinely analyzed by Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP-AES). Flame atomic absorption spectroscopy (FAAS) is also used routinely for the confirmation and the analysis of metals. Due to different sample preparation requirements, antimony, silver, mercury and hexavalent chromium are not included in the routine ICP-AES metal scan, so they should be specifically requested if they are suspected to be present in a sample. The laboratory should also be informed if a water sample requires drinking water detection limits for metals. Use of the heated graphite furnace atomic absorption spectrometer (HGA-AAS) for individual metals (tedious and time consuming) may be necessary to attain the low detection limits required for the

drinking water criteria. Overall, methods selection is dependent on the analyte and detection limit requirement.

2) Anions:

The anions listed in Table 4.1-16 are routinely analyzed by ion chromatography (IC). Cyanide or sulfide analysis (tedious and time consuming) is accomplished by an acid distillation of the sample, followed by a colorimetric determination of the distillate. Individual spot tests may also be requested to qualitatively screen for cyanide, nitrate, nitrite, fluoride, chloride, bromide, sulfate, sulfite and/or sulfides in a sample. Spot test results are generally reported as positive or negative for that anion.

3) Organolead:

Organolead samples require a solvent extraction workup, then flame AA analysis. The results are reported as total organolead. Speciation of tetraalkyl leads is currently not performed.

4) Hg Analyses:

By cold vapor spectrophotometric techniques.

5) Waste Extraction Test (WET):

The WET utilizes a 48-hour extraction with a citric acid buffer solution (pH 5) to determine the leachability of metals from solid wastes as described in Title 22, California Code of Regulation, Chapter 11, Article 5, Appendix II. However, a total metals analysis is normally requested and performed on a sample before the WET. The WET (if requested) is performed only when the total concentration of a toxic metal is less than its TTLC (Total Threshold Limit Concentration) value, but greater than 10 times its STLC (Soluble Threshold Limit Concentration) value.

Note: It not necessary to perform the WET for the toxic organic compounds, since their TTLC values are exactly 10 times their respective STLC values.

6) TCLP for Metals:

The TCLP utilizes an 18 $\pm$ 2 hour extraction with an acetic acid buffer solution (pH 2.88 or 4.93) to determine the leachability of metals from solid wastes as described in EPA Method 1311.

Note: ECL studies have indicated that the WET is more aggressive than the TCLP in leaching metals from solid waste.

4.1.3.4 Special Analysis Requests:

As mentioned previously, ECL is capable of analyzing and characterizing unknown solid or liquid samples that cannot be identified, quantified or characterized by routine analysis. Although resources are limited, ECL also has the expertise and instrumentation to develop and perform difficult, non-routine analyses that may not be available from commercial laboratories. The requestor should consult with laboratory prior to submitting a request for this service. Examples of non-routine analysis are:

1) Polychlorinated Dibenzodioxins and Dibenzofurans:

ECL has developed methods for the trace analysis of polychlorinated dibenzodioxins and dibenzofurans in environmental and biological samples. The analysis is performed by GC/MS and is toxic-isomer specific for the determination of 2,3,7,8-Tetrachlorodibenzodioxin (2378-TCDD) equivalents. Total congener group concentrations are also reported. Detection limits are at the parts per trillion range. Because the clean up procedures, instrument analysis and data processing are long and laborious, the analysis is expensive. Please contact the Trace Analysis Group (8-571-3624) for information about sample containers, current turn-around time estimates, or specifics of the analysis.

ECL has also developed a faster screening method for PCDD/Fs that is based on measuring the OCDD and OCDF concentrations by gas chromatography with electron capture detection. Its use is intended for those cases where a large number of samples must be taken to characterize the site and where OCDD and OCDF are appropriate surrogates for PCDD/Fs. Our current facilities with this screening method are limited, and its use will be made on a case by case basis.

2) Congener-specific PCBs

ECL has developed methods for the analysis of specific PCB congeners at trace levels. As opposed to the determination of PCBs as Aroclors, congener-specific analysis allows the measurement of individual PCBs. The widest application of such technique is for human and ecological risk assessment, where a few toxic PCB congeners are of special interest. The technique involves laborious sample cleanup and HRGC-HRMS analysis and it is time consuming and expensive. For information about the appropriateness and availability of this type of analysis, contact the Trace Analysis Group (8-571-3624).

Table 4.1-0 lists the WHO / International Toxic Equivalency Factors.

ISOMER	POTENCY FACTOR I-TEF
2,3,7,8-TCDD	1.0
1,2,3,7,8-PeCDD	1.0
1,2,3,4,7,8-HxCDD	0.1
1,2,3,6,7,8-HxCDD	0.1
1,2,3,7,8,9-HxCDD	0.1
1,2,3,4,6,7,8-HpCDD	0.01
OCDD	0.0001
2,3,7,8-TCDF	0.1
1,2,3,7,8-PeCDF	0.5
2,3,4,7,8-PeCDF	0.1
1,2,3,4,7,8-HxCDF	0.1
1,2,3,6,7,8-HxCDF	0.1
2,3,4,6,7,8-HxCDF	0.1
1,2,3,7,8,9-HxCDF	0.1
1,2,3,4,6,7,8-HpCDF	0.01
1,2,3,4,7,8,9-HpCDF	0.01
OCDF	0.0001

Table 4.1-0 TCDD Toxic Equivalent WHO/I-TEQ System for Dioxins, Furans, and dioxide-like PCBs.

4) Qualitative analysis of unknowns:

Qualitative analysis for unknowns may be performed by a variety of different techniques, depending on the sample. GC/MS, and Fourier transform infrared spectroscopy (FTIR) are some the available instrumentation employed by ECL to identify unknowns. Consult with ECL if this service is required.

5) Aquatic Toxicity Testing (Fish Bioassays:

A 96 hour LC_{50} aquatic toxicity testing using fathead minnows, rainbow trout or golden shiners is available as a special request and requires prior approval by ECL. The test is performed by ECL's contract laboratories. See Title 22, California Code of Regulations (5/31/91), 66261.24(a)(6) for the toxicity criteria.

Table 4.1-1. 8260B Volatile Organics

Acetone	CAS# 67-64-1	2,2-dichloropropane	594-20-7
Benzene	71-43-2	1,1-dichloropropene	563-58-6
Bromobenzene	108-86-1	cis-1,3-Dichloropropene	10061-01-5
Bromochloromethane	74-97-5	trans-1,3-Dichloropropene	10061-02-6
Bromodichloromethane	75.27-4	Ethylbenzene	100-41-4
Bromoform	75.25-2	Hexachlorobutadiene	87-68-3
Bromomethane	74-83-9	2-hexanone (MBK)	591-78-6
2-Butanone (MEK)	78-93-3	Isopropylbenzene	98-82-8
n-Butylbenzene	104-51-8	p-Isopropyltoluene	99-87-6
sec-Butylbenzene	135-98-8	methyl tertiary butyl ether **	16434-04-4
tert-Butylbenzene	98-06-6	4-methyl-2-pentanone(MIBK)	108-10-1
Carbon tetrachloride	56-23-5	Methylene chloride(Dichloromethane)	75-09-2
Chlorobenzene	108-90-7	Naphthalene	91-20-3
Chloroethane	75-00-3	N-propylbenzene	103-65-1
Chloroform	67-66-3	Styrene	100-42-5
Chloromethane	74-87-3	1,1,1,2-tetrachloroethane	630-20-6
2-chlorotoluene	95-94-8	1,1,2,2 Tetrachloroethane	79-34-5
4-chlorotoluene	106-43-4	Tetrachloroethene	127-18-4
Dibromochloromethane	124-48-1	Toluene	108-88-3
1,2-Dibromo-3-chloropropane**	96-12-8	1,2,3-Trichlorobenzene	87-61-6
1,2-Dibromoethane	106-93-4	1,2,4-Trichlorobenzene	120-82-1
Dibromomethane	74-95-3	1,1,1-Trichloroethane	71-55-6
1,2-Dichlorobenzene	95-50-1	1,1,2-Trichloroethane	79-00-5
1,3-Dichlorobenzene	541-73-1	Trichloroethene	79-01-6
1,4-Dichlorobenzene	106-46-7	Trichlorofluoromethane	75-69-4
Dichlorodifluoromethane	75-71-8	1,2,3-Trichloropropane	96-18-4
1,1-Dichloroethane	75-34-3	1,2,4-Trimethylbenzene	95-63-6
1,2-Dichloroethane	107-06-2	1,3,5-Trimethylbenzene	108-67-8
1,1-Dichloroethene	75-35-4	Vinyl chloride	75-01-4
cis-1,2-Dichloroethene	156-59-2	m & p-Xylene(s)	108-38-3
trans-1,2-Dichloroethene	156-60-5	0-Xylene	95-47-6
1,2-dichloropropane	78-87-5		
1,3-dichloropropane	142-28-9	** Determined by special request only.	

Table 4.1-2. 8270 Semivolatile Organics

Acenaphthene	CAS# 83-32-9	2,4-Dinitrotoluene	121-14-2
Acenaphthylene	208-96-8	2,6-Dinitrotoluene	606-20-2
Aniline	62-53-3	Di-n-octyl phthalate	117-84-0
Anthracene	120-12-7	Fluoranthene	206-44-0
Benzo(a)anthracene	56-55-3	Fluorene	86-73-7
Benzo(b)fluoranthene	205-99-2	Hexachlorobenzene	118-74-1
Benzo(k)fluoranthene	207-08-9	Hexachlorobutadiene	87-68-3
Benzo(g,h,i)perylene	191-24-2	Hexachlorocyclopentadiene	77-47-4
Benzo(a)pyrene	50-32-8	Hexachloroethane	67-72-1
Benzyl alcohol	100-51-6	Indeno(1,2,3-cd)pyrene	193-39-5
Bis(2-chloroethoxy)methane	111-91-1	Isophorone	78-59-1
Bis(2-chloroethyl) ether	111-44-4	2-Methylnaphthalene	91-57-6
Bis(2-chloroisopropyl) ether	108-60-1	2-Methylphenol	95-48-7
Bis(2-ethylhexyl) phthalate	117-81-7	4&3-Methylphenol	106-44-5
4-Bromophenyl phenyl ether	101-55-3	Naphthalene	91-20-3
Butyl benzyl phthalate	85-68-7	2-Nitroaniline	88-74-4
Carbazole	86-74-8	3-Nitroaniline	99-09-2
4-Chloroaniline	106-47-8	4-Nitroaniline	100-01-6
1-Chloronaphthalene	90-13-1	Nitrobenzene	98-95-3
2-Chloronaphthalene	91-58-7	2-Nitrophenol	88-75-5
4-Chloro-3-methylphenol	59-50-7	4-Nitrophenol	100-02-7
2-Chlorophenol	95-57-8	N-Nitrosodimethylamine **	62-75-9
4-Chlorophenyl phenyl ether	7005-72-3	N-Nitrosodiphenylamine	86-30-6
Chrysene	218-01-9	N-Nitrosodipropylamine	621-64-7
Dibenz(a,h)anthracene	53-70-3	Pentachlorophenol	87-86-5
Dibenzofuran	132-64-9	Phenanthrene	85-01-8
Di-n-butyl phthalate	84-74-2	Phenol	108-95-2
1,3-Dichlorobenzene	541-73-1	Pyrene	129-00-0
1,4-Dichlorobenzene	106-46-7	Pyridine ***	110-86-1
1,2-Dichlorobenzene	95-50-1	1,2,4-Trichlorobenzene	120-82-1
3,3'-Dichlorobenzidine	91-94-1	2,4,5-Trichlorophenol	95-95-4
2,4-Dichlorophenol	120-83-2	2,4,6-Trichlorophenol	88-06-2
Diethyl phthalate	84-66-2		
2,4-Dimethylphenol	105-67-9		
Dimethyl phthalate	131-11-3		
4,6-Dinitro-2-methylphenol	534-52-1		
2,4-Dinitrophenol	51-28-5		

** Determined by special request only
*** Determined if TCLP-Semivolatile Organics is requested

Table 4.1-3. 8082 Polychlorinated Biphenyls (PCBs)

Aroclor 1016	CAS # 12674-11-2	Aroclor 1248	12672-29-6
Aroclor 1221	11104-28-2	Aroclor 1254	11097-69-1
Aroclor 1232	11141-16-5	Aroclor 1260	11096-82-5
Aroclor 1242	53469-21-9	Aroclor 1262	37324-23-5

4.1-4. 8081 Organochlorine Pesticides

delta-BHC	CAS # 319-86-8	Dieldrin	60-57-1
alpha-BHC	319-84-6	o,p'-DDD **	53-19-0
beta-BHC	319-85-7	Endrin	72-20-8
gamma-BHC (Lindane)	58-89-9	Endrin aldehyde	7421-93-4
PCNB **	82-68-8	Endosulfan sulfate	1031-07-8
Heptachlor	76-44-8	Endosulfan II	33213-65-9
Aldrin	309-00-2	p,p'-DDD	72-54-8
Heptachlor epoxide	1024-57-3	o,p'-DDT **	789-02-6
alpha-Chlordane	5103-71-9	p,p'-DDT	50-29-3
o,p'-DDE **	3424-82-6	p,p'-Methoxychlor	72-43-5
Endosulfan I	959-98-8	Tedion **	116-29-0
gamma-Chlordane	5103-74-2	Mirex	2385-85-5
p,p'-DDE	72-55-9	Toxaphene	8001-35-21

** Note: Included only by special request.

Table 4.1-5. 8141A Organophosphorus Pesticides

Dichlorvos (DDVP)	CAS # 62-73-7	Fonofos (Dyfonate)	944-22-9
Demeton-O	8065-48-3	Disulfoton (DisySMOn)	298-04-4
Phorate (Thimet)	298-02-2	Demeton-S	8065-48-3
Ethoprop (Mocap)	13194-48-4	Chlorpyrifos	2921-88-2
Diazinon	333-41-5	Dimethoate **	60-51-5
Ronnel	299-84-3	Monocrotophos **	6923-22-4
Fenthion (Baytex)	55-38-9	Parathion, methyl	298-00-0
Tokuthion **	34643-46-4	Malathion	121-75-5
DEF (Butifos) **	78-48-8	Chlorfenvinphos **	470-90-6
Parathion, ethyl	56-38-2	Methidathion **	950-37-8
Trithion	786-19-6	Ethion	563-12-2
Phospholan **	947-02-4	Leptophos	21609-90-5
Fensulfothion **	115-90-2	EPN	2104-64-5
Phosmet	732-11-6	Azinphos, methyl	86-50-0
Azinphos, ethyl	2642-71-9	Famphur **	52-85-7
Mevinphos	7786-34-7	Coumaphos	56-72-4
Sulfotepp **	3689-24-5		

** Note: Included only by special request.

Table 4.1-6. 8151A Chlorinated Herbicides

2,4-D	CAS # 94-75-7	Dichlorprop	120-36-5
2,4,5-T	93-76-5	MCPA	94-74-6
2,4-DB	94-82-6	Dalapon	75-99-0
2,4,5-TP (Silvex)	93-72-1	Dinoseb	88-85-7
Dicamba	1918-00-9	MCPP	93-65-2

Table 4.1-7. 8310 Polynuclear Aromatic Hydrocarbons (PAHs or PNAs)

Naphthalene	CAS # 91-20-3	Benzo(a)anthracene	56-55-3
Acenaphthylene	208-96-8	Chrysene	218-01-9
Acenaphthene	83-32-9	Benzo(b)fluoranthene	205-99-2
Fluorene	86-73-7	Benzo(k)fluoranthene	207-08-9
Phenanthrene	85-01-8	Benzo(a)pyrene	50-32-8
Anthracene	120-12-7	Dibenzo(ah)anthracene	53-70-3
Fluoranthene	206-44-0	Benzo(ghi)perylene	191-24-2
Pyrene	129-00-0	Indeno(1,2,3-cd)pyrene	193-39-5

Table 4.1-8. 8330 Nitroaromatics and Nitramines

Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	CAS# 2691-41-0
Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)	121-82-4
1,3,5-Trinitrobenzene (1,3,5-TNB)	99-35-4
Methyl 1-2,4,6-trinitrophenylnitramine (Tetryl)	479-45-8
Nitrobenzene (NB)	98-95-3
2,4,6-Trinitrotoluene (2,4,6-TNT)	118-96-7
4-Amino-2,6-dinitrotoluene (4-Am-DNT)	1946-51-0
2-Amino-4,6-dinitrotoluene (4-Am-DNT)	355-72-78-2
2,4-Dinitrotoluene (2,4-DNT)	121-14-2
2,6-Dinitrotoluene (2,6-DNT)	606-20-2
2-Nitrotoluene (2-NT)	88-72-2
3-Nitrotoluene (3-NT)	99-08-1
4-Nitrotoluene (4-NT)	99-99-0

Table 4.1-9. 734.1 N-Methylcarbamate Pesticides

Aldicarb sulfone	CAS # 1646-88-4	Propoxur (Baygon)	114-26-1
Methomyl (Lannate)	16752-77-5	Carbofuran (Furadan)	1563-66-2
3-Hydroxycarbofuran	16655-82-6	Carbaryl (Sevin)	63-25-2
Dioxacarb	6988-21-2	Methiocarb (Mesurol)	2032-65-7
Aldicarb	116-06-3	Promecarb	2631-37-0

Table 4.1-10. 736 Dinitro-Compounds and Dinoseb

4-Nitrophenol	CAS # 100-02-7	Dinoseb	88-85-7
2,6-Dinitrophenol	573-56-8	Dinitramine (Cobex)	29091-05-2
2,4-Dinitrophenol	51-28-5	Fluchoralin (Basalin)	33245-39-5
3,4-Dinitrophenol	557-71-9	Pendimethalin (Prowl)	40487-42-1
2,5-Dinitrophenol	329-71-5	Trifluralin (Treflan)	1582-09-8
2-Methyl-4,6-dinitrophenol	534-52-1	Dinocap (I and II)	39300-45-3
4,6 Dinitro-o-cresol	121-14-2		

Table 4.1-11. TCLP Volatile Organics with GCMS Method 8260

Benzene	CAS # 71-43-2	1,1-Dichloroethylene	75-35-4
Chlorobenzene	108-90-7	2-Butanone (MEK)	78-93-3
Chloroform	67-66-3	Tetrachloroethene (Tetrachloroethylene)	127-18-4
1,4-Dichlorobenzene	106-46-7	Trichloroethene (Trichloroethylene)	79-01-6
Carbon tetrachloride	56-23-5	Vinyl chloride	75-01-4
1,2-Dichloroethane	107-06-2		

Table 4.1-12. TCLP Semivolatile Organics with GCMS Method 8270

1,4-Dichlorobenzene	CAS # 106-46-7	3-Methylphenol (m-cresol)*	108-39-4
2,4-Dinitrotoluene	121-14-2	4-Methylphenol (p-cresol)*	106-44-5
Hexachlorobenzene	118-74-1	Nitrobenzene	98-95-3
Hexachlorobutadiene	87-68-3	Pentachlorophenol	87-86-5
Hexachloroethane	67-72-1	Pyridine	110-86-1
2-Methylphenol(0-cresol)	95-48-7	2,4,5-Trichlorophenol	95-95-4
		2,4,6-Trichlorophenol	88-06-2

* Compounds coelute - reported as M & P cresol

Table 4.1-13. TCLP Chlorinated Pesticides

Chlordane	CAS # 12789-03-6	Lindane	58-89-9
Heptachlor	76-44-8	Methoxychlor	72-43-5
Endrin	72-20-8	Toxaphene	8001-35-21

Table 4.1-14. TCLP Herbicides

2,4-D	CAS # 94-75-7	2,4,5-TP (Silvex)	93-72-1

Table 4.1-15 EPA 6010B Metals by ICP-AES

As-Arsenic	Ni-Nickel
Ba-Barium	Pb-Lead
Be-Beryllium	Se-Selenium
Cd-Cadmium	Tl-Thallium
Co-Cobalt	V-Vanadium
Cr-Chromium	Zn-Zinc
Cu-Copper	
Mo-Molybdenum	

Table 4.1-16 - Anions Method ECL 960

Fluoride	Nitrate
Chloride	Phosphate
Sulfate	

Table 4.1-21: LIST OF ANALYTICAL METHODS - EPA, ECL, ECL-SC and Contract Labs

Organic Methods

NAME OF METHOD	EPA	ECL	ECL-SC	CONTRACT
4-Chlorobenzene sulfonic acid by Ion Chromatography	_____	732[a] (5)	_____	_____
N-Methylcarbamates	8318 (15)	8318 (15)	8318 (15)	_____
Nitroaromatics & Nitramines by HPLC	8330 (17)	8330 (17)	_____	_____
4,4'-Methylenebis(2-chloroaniline) (MOCA)	_____	740 (5)	_____	_____
Polynuclear Aromatic Hydrocarbons by HPLC	8310 (1)	8310 (1)	8310 (1)	8310 (1)
Volatile Organic Analysis by GC/PID & ELCD	8021 B (17)	8021 B (17)	8021 B (17)	8021 B (17)
Volatile Organosulfur Compounds	_____	760 (5)	_____	_____
Volatile Hydrocarbon Analysis by GC/FID	_____	815 (5)	815 (5)	8015 B (16)
1,2-Dibromoethane(EDB) and 1,2-Dibromo-3-chloropropane(DBCP)	8011 (2)	8011[b] (2)	8011[b] (2)	_____
Diesel Analysis by GC/FID	8015B (16)	8015B (16)	8015B (16)	8015 B (16)
Phenols by GC/FID	8041 (16)	8041 (16)	8041 (16)	8041A(16)
Chlorophenols by Acetylation and GC/ECD	_____	782 (5)	782 (5)	_____
Base & Neutral Extractable Organics by GC/FID	8015 B(16)	8270C (2)	8270 C (2)	8015B(16)
Polynuclear Aromatic Hydrocarbons	8100 (1)	8100 (1)	8100 (1)	8100 (1)
Organochlorine Pesticides	8081A (16)	8081A (16)	8081A (16)	8081A (16)
PCBs	8082 (16)	8082 (16)	8082 (16)	8082 (16))
Organophosphorus Pesticides (Capillary)	8141A(15)	8141A (15)	_____	8141A(15)
Chlorinated Herbicides	8151 A (16)	8151 A (16)	8151 A (16)	8150A (16)
GC/MS Confirmation of Base Neutral & Acid Extractable Organics	_____	835[a] (5)	8270 (2)	_____
GC/MS Method for Volatile Organics: Capillary Column Technique	8260 B (16)	8260B (16)	8260B (16)	_____
GC/MS Method for Semivolatile Organics: Capillary Column Technique	8270 D (17)	8270 A (2)	8270 A (2)	8270 C (16)
GC/MS Headspace Method for Volatile Organics	_____	850 (5)	850 (5)	_____
Screening for Dithiocarbamate Residues in Soil by Headspace Analysis of CS^2	_____	820 (5)	_____	_____
GC/NPD Screening Procedures EPTC Screening in Soil	_____	821 (5)	_____	_____
Malathion and Related Products	_____	841 (5)	_____	_____
OCCC and OCDF by Electron Capture GLC	_____	895 (5)	_____	_____
Analysis of PCDDs and PCDFs by GC/MS; Environmental Samples (Soxhlet)	8280B (17)	880 (5)	_____	8290(15)
Analysis of PCDDs and PCDFs by GC/HRMS; Biological Samples (Stalling)	8290A (17)	880 (5)	_____	8290 (15)
Total Organic Halides	9020 B (15)	9020 A (2)	_____	_____
Total Recoverable Oil & Grease (Gravimetric, Separatory Funnel Extraction)	1664(15)	9070 (1)	9070 (1)	_____
Oil & Grease Extraction Method for Sludge Samples	9071 B (15)	9071 (1)	9071 (1)	_____
Total Recoverable Oil and Grease by Infrared	413.2 (3)	413.2 (3)	413.2 (3)	_____
Total Recoverable Petroleum Hydrocarbons by Infrared	418.1 (3)	418 (3)	418.1 (3)	_____
Total Halogens in Oil	_____	792 (5)	_____	_____

Inorganic Methods

Acid Digestion of Water for Total Recoverable or Dissolved Metals	3005 A (2)	3005 A (2)	_____	_____
Acid Digestion of Aqueous Samples and Extracts for Totals Metals	3010 A (2)	3010 A (2)	_____	_____
Digestion of Solids for Metal Determinations	3050 B (16)	3050 A (2)	3.23 (6)	3050 A (2)
Inductively Coupled Plasma Atomic Emission Spectrometry (ICPAES) 16 metals	6010 C (18)	6010 A (2)	6010 A (2)	6010 B (16)
Inductively Coupled Plasma - Mass Spectrometry	6020 A (17)	6020[b] (10)	_____	_____
Antimony (AA, Direct Aspiration)	7040 (1)	7040 (1)	7040 (1)	7040 (1)
Antimony (AA, Furnace Technique)	7041 (1)	7041 (1)	7041 (1)	7041 (1)
Arsenic (AA, Flame)	7060 A (15)	7060 (1)	_____	7060 A (15)
Arsenic (AA, Furnace Technique)	7061 A (2)	7061 A (2)	7061 A (2)	7061 A (2)
Arsenic (AA, Gaseous Hydride)	7080 A (15)	7080 (1)	7080 (1)	7080 A (15)
Barium (AA, Direct Aspiration)	7081 (2)	7081 (2)	7081 (2)	7081 (2)
Barium (AA, Furnace Technique)	7090 (1)	7090 (1)	7090 (1)	7090 (1)
Beryllium (AA, Direct Aspiration)	7091 (1)	7091 (1)	7091 (1)	7091 (1)
Beryllium (AA, Furnace Technique)	7130 (1)	7130 (1)	7130 (1)	7130 (1)
Cadmium (AA, Direct Aspiration)	7131 A (15)	7131 (1)	7131 (1)	7131 A (15)

NAME OF METHOD EPA ECL ECL-SC CONTRACT

Cadmium (AA, Furnace Technique)	7190 (1)	7190 (1)	7190 (1)	7190 (1)
Chromium (AA, Direct Aspiration)	7191 (1)	7191 (1)	7191 (1)	7191 (1)
Chromium (AA, Furnace technique)	7195 (1)	_____	7195 (1)	7195 (1)
Chromium, Hexavalent (Coprecipitation)	7196 A (2)	7196 A (2)	_____	7196 A (2)
Chromium, Hexavalent (Colorimetric)	7197 (1)	_____	7197 (1)	7197 (1)
Chromium, Hexavalent (Chelation/Extraction)	7200 (1)	7200 (1)	7200 (1)	7200 (1)
Cobalt (AA, Direct Aspiration)	7201 (1)	7201 (1)	7201 (1)	7201 (1)
Cobalt (AA, Furnace Technique)	7210 (1)	7210 (1)	7210 (1)	7210 (1)
Copper (AA, Direct Aspiration)	7211 (2)	7211 (2)	7211 (2)	7211 (2)
Copper (AA, Furnace Technique)	7420 (1)	7420 (1)	7420 (1)	7420 (1)
Lead (AA, Direct Aspiration)	7421 (1)	7421 (1)	7421 (1)	7421 (1)
Lead (AA, Furnace technique)	7470 A (15)	7470 (1)	7470 (1)	7470 A (15)
Mercury in Liquid Waste (Manual Cold Vapor Technique)	7471 B (17)	7471 (1)	7471 (1)	7471 A (16)
Mercury in Solid and Semisolid Waste (Manual Cold Vapor Technique)	7480 (1)	7480 (1)	7480 (1)	7480 (1)
Molybdenum (AA, Direct Aspiration)	7481 (1)	7481 (1)	7481 (1)	7481 (1)
Molybdenum (AA, Furnace Technique)	7520 (1)	7520 (1)	7520 (1)	7520 (1)
Nickel (AA, Direct Aspiration)	7740 (1)	7740 (1)	7740 (1)	7740 (1)
Selenium (AA, Furnace Technique)	7741 A (15)	7741 (1)	7741 (1)	7741 A (15)
Selenium (AA, Gaseous Hydride)	7760 A (2)	7760 A (2)	7760 A (2)	7760 A (2)
Silver (AA, Direct Aspiration)	7761 (2)	7761 (2)	7761 (2)	7761 (2)
Silver (AA, Furnace Technique)	7840 (1)	7840 (1)	7840 (1)	7840 (1)
Thallium (AA, Direct Aspiration)	7841 (1)	7841 (1)	7841 (1)	7841 (1)
Thallium (AA, Furnace Technique)	7910 (1)	7910 (1)	7910 (1)	7910 (1)
Vanadium (AA, Direct Aspiration)	7911 (1)	7911 (1)	7911 (1)	7911 (1)
Vanadium (AA, Furnace Technique)	7950 (1)	7950 (1)	7950 (1)	7950 (1)
Zinc (AA, Direct Aspiration)	7951 (2)	7951 (2)	7951 (2)	7951 (2)
Zinc (AA, Furnace Technique)	_____	938 (5)	_____	_____
Organolead by FAAS	9056 A (18)	300.0 (7)	300.0 (7)	300.0 (7)
Ion Chromatography (F, Cl, NO_3, NO_2, SO_4, Br, PO_4)	_____	965[a] (5)	965[a] (5)	_____
Ion Selective Electrode Method (F, Cl, CN)	_____	970[a] (5)	970[a] (5)	340 (3)
Fluoride	9214 (1)	9251 (1)	_____	325 (3)
Chloride (Colorimetric, Automated Ferricyanide AAII)	9251 (1)	_____	_____	_____
Chloride (Titrimetric, Mercuric Nitrate)	_____	_____	_____	300 (3)
Nitrate	9210A(18)	976[a] (5)	976[a] (5)	300 (3)
Nitrite	354.1	_____	9035 (1)	375 (3)
Sulfate (Colorimetric, Automated Methylthymol)	9036 (1)	9038 (1)	_____	375 (3)
Sulfate (Turbidimetric)	9038 (2)	990 (5)	9010 (1)	9010B(16)
Cyanide	9213 (16)	376.2 (3)	9030 (1)	9215(16)
Sulfide	9215(16)	_____	_____	_____

Miscellaneous Methods

Pensky-Martens Closed-Cup Method for Determining Ignitability	1010 (1)	_____	_____	1010 (1)
Setaflash Closed-Cup Method for Determining Ignitability	1020 A (2)	1020 A (2)	1020 A (2)	1020 A (2)
Flammability of Compressed Gases (Aerosol Products)	_____	720 (14)(5)	_____	_____
Determination of % Dry Solids	(1)	704-S (1)(5)	704-S (1)(5)	(1)
pH Electrometric Measurement	9040B (15)	9040 (1)	9040 (1)	9040 B (15))
Soil pH	9045 C (15)	9045 A (2)	9045 A (2)	9040B(15)
Specific Conductance	9050 A (16)	9050 (1)	9050 (1)	_____
Total Organic Carbon	9060 (1)	_____	_____	_____
California Waste Extraction Test (WET)	_____	910 (5)	910 (5)	66700 (8)
EP Toxicity Extraction	1310 A (2))	1310 (1)	1310 (1)	1310A(2)
Toxicity Characteristic Leaching Procedure (TCLP)	1311(2)	1311 (2)	1311(2)	1311 (2)

Miscellaneous Methods Other Laboratories

NAME OF METHOD	EPA	ECL	ECL-SC	CONTRACT/ AIHL/SRL
Fish Bioassay	_____	66696 (8)	_____	66261.24(a)(16)
Corrosivity	_____	66708 (8)	_____	66261.22
Reactivity	_____	66705 (8)	_____	66261.23)
Asbestos	_____	_____	_____	Appendix IV, Table 4,
Acidity	305.1 (3)	_____	_____	305.1 (3)
Alkalinity	310.1 (3)	_____	_____	310.1 (3)
Chemical Oxygen Demand	410.1 (3)	_____	_____	410.1 (3)
Soil Gas	_____	_____	_____	TO 14A(MS)

Notes: [a] Analyses performed, method not available in SOP format.
[b] Planned for future; projected date: XX-XX-90.
[c] Planned for future; projected date: XX-XX-90.

References:

1. Test Methods for Evaluating Solid Waste; Physical/Chemical Methods, SW846, U.S.EPA, 3rd edition, September 1986.
2. Update I, July 1992, for SW-846, 3rd edition.
3. Methods for Chemical Analysis of Water and Wastes, EPA600/479020, Revised March 1983.
4. Method 531. Measurement of N-Methyl Carbamoyloximes and N-Methyl carbamates in Drinking Water by Direct Aqueous Injection HPLC with Post Column Derivatization, EPA/600/485/054.
5. ECL Method.
6. ECL-SC Method.
7. Method 300.0, EPA600/484017 series.
8. California Administrative Code, Title 22, Chapter 30, Article 11, "Criteria for Identification of Hazardous and Extremely Hazardous Waste".
9. Method 632, The determination of Carbamate and Urea pesticides in industrial and municipal waste, EPA600/482014.
10. Draft Methods.
11. EPA Draft Methods for Drinking Water.
12. Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, EPA600/482057.
13. EPA Draft Methods for Wastewater.
14. ASTM Method
15. Update II B, September 1994, for SW-846, 3rd Edition.
16. Update III, December 1996, for SW-846, 3rd Edition.
17. Update IVA, for SW-846
18. Update IVB, for SW-846

Table 4.1-22: LIST OF ANALYTICAL METHODS - EPA Equivalent Methods

Organic Methods

NAME OF METHOD	SOLID WASTE	DRINKING WATER	WASTEWATER
Volatile Organic Halides	_____	502.1 (11)	601 (12)
1,2-Dibromoethane and 1,2-Dibromo-3-Chloropropane	8011 (2)	504 (11)	_____
Non-Halogenated Volatile Organics	8015 C (18)	_____	_____
Aromatic Volatile Organics	_____	503.1 (11)	602 (12)
Volatile Organic Compounds in by Purge-and-Trap Capillary Column GC	8021 B (16)	502.2 (11)	_____
Acrolein, Acrylonitrile, Acetonitrile	_____	_____	603 (12)
Phenols by GC/FID and ECD confirmation	_____	_____	604 (12)
Benzidine	…	_____	605 (12)
Phthalate Esters	8061 A(16)	_____	606 (12)
Nitrosamines	8070 A (16)	_____	607 (12)
Organochlorine Pesticides and PCBs	_____	_____	608 (12)
Organochlorine Pesticides and PCBs	8081 B(18))	508 (11)	_____
Nitroaromatics and Cyclic Ketones	8091 (16)	_____	609 (12)
Polynuclear Aromatic Hydrocarbons by GC/FID	8100 (1)	_____	610(12)
Haloethers	8111 (16)	_____	611 (12)
Chlorinated Hydrocarbon	8121 (14)	_____	612 (12)
Organophosphorus Pesticides	_____	_____	614(12)
Organophosphorus Pesticides Capillary Column	8141 B (17)	507 (11)	_____
Chlorinated Herbicides	8151A (16)	515 (11)	615(12)
GC/MS Method for Volatile Organics: Packed Column Technique	_____	524.1 (11)	624 (12)
GC/MS Method for Semivolatile Organics: Packed Column Technique	_____	_____	625 (12)
GC/MS Method for Semivolatile Organics: Capillary Column Technique	8260 B (16))	524.2 (11)	_____
GC/MS Method for Volatile Organics: Capillary Column Technique	8270 D (17)	_____	_____
The Analysis of PCDDs and PCDFs	8280 B (17))	_____	_____
PCDDs and PCDFs by HRGC/HRMS	8290 A(17)	_____	_____
Carbamates	_____	531 (4)	632 (9)
Polynuclear Aromatic Hydrocarbons by HPLC	8310 (1)	_____	610 (12)
VOC by Isotope Dilution	_____	_____	1624(12)
SVOC by Isotope Dilution GC/MS	_____	_____	1625(12)

Inorganic Methods

Inductively Coupled Plasma Atomic Emission Spectrometry (ICPAES) (16 metals)	6010 C (18)	200.7 (3)	200.7 (3)
Aluminum (AA, Direct Aspiration)	7020 (2)	202.1 (3)	202.1 (3)
Antimony (AA, Direct Aspiration)	7040 (1)	204.1 (3)	204.1 (3)
Antimony (AA, Furnace Technique)	7041 (1)	204.2 (3)	204.2 (3)
Arsenic (AA, Furnace Technique)	7060 A (14)	206.2 (3)	206.2 (3)
Arsenic (AA, Gaseous Hydride)	7061 A (2)	206.3 (3)	206.3 (3)
Barium (AA, Direct Aspiration)	7080 A (14)	208.1 (3)	208.1 (3)
Beryllium (AA, Direct Aspiration)	7090 (1)	210.1 (3)	210.1 (3)
Beryllium (AA, Furnace Technique)	7091 (1)	210.2 (3)	210.2 (3)
Cadmium (AA, Direct Aspiration)	7130 (1)	213.1 (3)	213.1 (3)
Cadmium (AA, Furnace Technique)	7131 A (14)	213.2 (3)	213.2 (3)
Chromium (AA, Direct Aspiration)	7190 (1)	218.1 (3)	218.1 (3)
Chromium (AA, Furnace technique)	7191 (1)	218.2 (3)	218.2 (3)
Chromium, Hexavalent (Coprecipitation)	7195 (1)	218.5 (3)	218.5 (3)
Chromium, Hexavalent (Colorimetric)	7196 A (2)	_____	_____
Chromium, Hexavalent (Chelation/Extraction)	7197 (1)	218.4 (3)	218.4 (3)
Chromium, Hexavalent (Differential Pulse Polarography)	7198 (1)	_____	_____
Cobalt (AA, Direct Aspiration)	7200 (1)	219.1 (3)	219.1 (3)
Cobalt (AA, Furnace Technique)	7201 (1)	219.2 (3)	219.2 (3)
Copper (AA, Direct Aspiration)	7210 (1)	220.1 (3)	220.1 (3)
NAME OF METHOD	SOLID WASTE	DRINKING	WASTEWATER

NAME OF METHOD	SOLID WASTE	DRINKING WATER	WASTEWATER
Iron (AA, Direct Aspiration)	7380 (2)	236.1 (3)	236.1 (3)
Lead (AA, Direct Aspiration)	7420 (1)	239.1 (3)	239.1 (3)
Lead (AA, Furnace Technique)	7421 (1)	239.2 (3)	239.2 (3)
Lithium (AA, Direct Aspiration)	7430 (2)	_____	_____
Magnesium (AA, Direct Aspiration)	7450 (1)	242.1 (3)	242.1 (3)
Manganese (AA, Direct Aspiration)	7460 (1)	243.1 (3)	243.1 (3)
Mercury in Liquid Waste (Manual Cold Vapor Technique)	7470 A (14)	245.1 (3)	245.1 (3)
Mercury in Solid and Semisolid Waste (Manual Cold Vapor Technique)	7471B (17)	_____	_____
Molybdenum (AA, Direct Aspiration)	7480 (1)	246.1 (3)	246.1 (3)
Molybdenum (AA, Furnace Technique)	7481 (1)	246.2 (3)	246.2 (3)
Nickel (AA, Direct Aspiration)	7520 (1)	249.1 (3)	249.1 (3)
Osmium (AA, Direct Aspiration)	7550 (2)	252.1 (3)	252.1 (3)
Potassium (AA, Direct Aspiration)	7610 (2)	258.1 (3)	258.1 (3)
Selenium (AA, Furnace Technique)	7740 (1)	270.2 (3)	270.2 (3)
Selenium (AA, Gaseous Hydride)	7741 A (14)	270.3 (3)	270.3 (3)
Silver (AA, Direct Aspiration)	7760 A (2)	272.1 (3)	272.1 (3)
Sodium (AA, Direct Aspiration)	7770 (1)	273.1 (3)	273.1 (3)
Strontium (AA, Direct Aspiration)	7780 (2)	_____	_____
Thallium (AA, Direct Aspiration)	7840 (1)	279.1 (3)	279.1 (3)
Thallium (AA, Furnace Technique)	7841 (1)	279.2 (3)	279.2 (3)
Tin (AA, Direct Aspiration)	7870 (1)	282.1 (3)	282.1 (3)
Vanadium (AA, Direct Aspiration)	7910 (1)	286.1 (3)	286.1 (3)
Vanadium (AA, Furnace Technique)	7911 (1)	286.2 (3)	286.2 (3)
Zinc (AA, Direct Aspiration)	7950 (1)	289.1 (3)	289.1 (3)
Ion Chromatography (F, Cl, NO_3, NO_2, SO_4, Br, PO_4)	9056 A (18)	300.0 (7)	300.0 (7)
Sulfate (Colorimetric, Automated, Chloranilate)	9035 (1)	375.1 (3)	375.1 (3)
Sulfate (Colorimetric, Automated, Methylthymol Blue AA II)	9036 (1)	375.2 (3)	375.2 (3)
Sulfate (Turbidimetric)	9038 (1)	375.4 (3)	375.4 (3)
Total Amenable Cyanide (Colorimetric, Manual)	9010 B (16)	335.2 (3)	335.2 (3)
Total Amenable Cyanide (Colorimetric, Automated UV)	9012 A (16)	335.3 (3)	335.3 (3)
Total Organic Halides (TOX)	9020 B (14)	_____	_____
Purgeable Organic Halides (POX)	9021 (2)	_____	_____
Total Organic Halides (TOX) by Neutron Activation Analysis	9022 (1)	_____	_____
Acid-Soluble and Acid-Insoluble Sulfides	9030 B (6)	_____	_____
Extractable Sulfides	9031 (2)	376.1 (3)	376.1 (3)
Total Organic Carbon	9060 (1)	415.1 (3)	415.1 (3)
Phenolics (Spectrophotometric, Manual 4-AAP	9065 (1)	420.1 (3)	420.1 (3)
Phenolics (Colorimetric, Automated, 4-AAP	9066 (1)	420.2 (3)	420.2 (3)
Phenolics (Spectrophotometric, MBTH with Distillation)	9067 (1)	420.3 (3)	420.3 (3)
Total Recoverable Oil & Grease (Gravimetric, Separatory Funnel Extraction)	9070 (1)	413.1 (3)	413.1 (3)
Oil & Grease Extraction Method for Sludge Samples	9071 A (14)	_____	_____
Total Recoverable Oil and Grease by Infrared	_____	413.2 (3)	413.2 (3)
Total Recoverable Petroleum Hydrocarbons by Infrared	_____	418.1 (3)	418.1 (3)
Nitrate	_____	354 (3)	354 (3)
Nitrite	_____	352 (3)	352 (3)
Fluoride	_____	340 (3)	340 (3)
Chloride (Colorimetric, Automated Ferricyanide AAI)	9250 (1)	325.1 (3)	325.1 (3)
Chloride (Colorimetric, Automated Ferricyanide AAII)	9251 (1)	325.2 (3)	325.2 (3)
Chloride (Titrimetric, Mercuric Nitrate)	_____	325.3 (3)	325.3 (3)
Chloride (Titrimetric, Silver Nitrate)	9253 (14)	_____	_____

Miscellaneous Methods			
Pensky-Martens Closed-Cup Method for Determining Ignitability	1010 (1)	_____	_____
Setaflash Closed-Cup Method for Determining Ignitability	1020 A (2)	_____	_____
Extraction Procedure (EP) Toxicity Test	1310 A (2)	_____	_____
Acidity	_____	305.1 (3)	305.1 (3)
Alkalinity	_____	310.1 (3)	310.1 (3)
pH Electrometric Measurement	9040 B (14)	150.1 (3)	150.1 (3)
Soil pH	9045 C (2)	_____	_____
Specific Conductance	9050 A(16)	120.1 (3)	120.1 (3)
Chemical Oxygen Demand	_____	410.1 (3)	410.1 (3)

References

1. Test Methods for Evaluating Solid Waste; Physical/Chemical Methods, SW-846, U.S.EPA, 3rd edition, September 1986.
2. Update I, July 1992, for SW-846, 3rd edition.
3. Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, Revised March 1983.
4. Method 531. Measurement of N-Methyl Carbamoyloximes and N-Methyl carbamates in Drinking Water by Direct Aqueous Injection HPLC with Post Column Derivatization, EPA/600/4-85/054.
5. ECL Method.
6. ECL-SC Method.
7. Method 300.0, EPA-600/4-84-017 series.
8. California Administrative Code, Title 22, Chapter 30, Article 11, "Criteria for Identification of Hazardous and Extremely Hazardous Waste".
9. Method 632, The determination of Carbamate and Urea pesticides in industrial and municipal waste, EPA-600/4-82-014.
10. Draft Methods.
11. EPA Draft Methods for Drinking Water.
12. Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater, EPA-600/4-82-057. http://www.epa.gov/ostwater/Tools/guide/methods.html
13. EPA Draft Methods for Wastewater.
14. Update IIB, Sept. 1994, for SW-846, 3rd Edition

TABLE 4.1-23

ECL IN-HOUSE METHODS

Method#	Title of Method	Status (date completed)			
		Draft	Operational	Validated	Archived
3.24-M	Acid Digestion of Sediment, Sludge, and Soil				4/18/90
418-M (SCL)	Total Petroleum Hydrocarbons Spectrophotometric, Infrared				8/91
705-M	Analysis of Particulates not Otherwise Regulated, Total				1/11/96
720-M	**Flammability of Compressed Gasses (Aerosol Products)**				**9/12/02***
732-M	4-Chlorobenzene Sulfonic Acid by Ion Chromatography	pending			
736-M	**Dinitroaromatics by HPLC**				**6/18/03***
740-M	4,4'-Methylenebis(2-Chloraniline)-MOCA				2/10/98
750-M	**Determination of Polybromodiphenyl Ethers in E-Waste and Solid Matrices by Gas Chromatography**		**10/16/03***		
760-M	**Volatile Organosulfur Compounds**				**3/15/01***
768-M	Diesel by GC/FID				4/7/98
772-M	**2-Butoxyethanol in Water by GC/FID**		**9/9/03***		
782-M	**Chlorophenols by Acetylation and GC/ECD**				**9/9/03**
795-M	Determination of Chlorobiphenyls				4/23/91
800-M (SCL)	Acid Digestion of Oils for ICP and FAA	pending			
815-M (SCL)	**Gasoline and Gasoline Range Organics (GRO) by GC/FID Purge and Trap Method with Optional BTXE Analysis and PID in series**		**6/10/04***		
816-M (SCL)	**Diesel Range (DRO) and Motor Oil Range (MORO) Organics by GC/FID**		**6/10/04***		

Method#	Title of Method	Status (date completed)			
		Draft	Operational	Validated	Archived
820-M	**Screening for Dithiocarbamate Residues in Soil by Headspace Analysis of Carbon Disulfide**				**5/12/99***
821-M	**GC/NPD Screening Procedures EPTC Screening in Soil**				**5/12/99***
822-M	**Determination of Carbendazim and Thiophanate-Methyl in Soil**				**6/10/98***
841-M	Malathion and Related Products				4/15/98
850-M	Methylisothiocyanate in Vegetation Samples by GC/MS Capillary Column Purge and Trap		pending		
880-M	Polychlorinated Dibenenzo-P-Dioxins and Polychlorinated Dibenzofurans	5/92		pending	
895-M	OCDD and OCDF by Electron Capture "GLC"				9/28/89
938-M	**Determination of Organic Lead Compounds by FAAS**	**6/10/03***			
939-M	**Determination of Organic Lead Compounds by Graphite FAAS**		**4/16/04***		
940-M	Soluble and Total Phosphorus Analysis by ICP				9/9/97
973-M	Determination of Leached Chlorine from Solid Samples				10/9/97
3031-M	Acid Digestion of Organic Materials for ICP-AES and FAAS				9/94

*****Method signed

TABLE 4.1-24

ECL SOPs

SOP #	Title of SOP	Status (date completed)		
		Draft	**Final**	**Archived**
600-S	Ahuraa (First Defender) Raman Spectroscopy	pending		
700-S	**General Glassware Preparation**		**4/12/04***	
701-S	**Initial Sample Preparation for Volatile Organic Analysis**		**3/4/05***	
702-S	**Initial Sample Preparation for Semi-Volatile Organic and Inorganic Samples**		**3/4/05***	
703-S	**Initial Sample Prep. for Samples not Requiring Waste Characterization**		**3/4/05***	
704-S	**Operation and Cleaning of Automated Milling Equipment**		**9/21/05***	
705-S	**Operation of Sample Ejector**		**9/21/05***	
706-S	**Sample Receipt**		**3/22/05***	
707-S	**Sample Handing and Chain-of-Custody**		**7/27/04***	
708-S	**Management of Lab. Generated Hazardous Materials**		**5/14/03***	
709-S	**Sample Storage and Disposal**		**4/29/05***	
710-S	Records Management	pending		
711-S	**Protocol for the Disposal of ECL Samples Analyzed by Commercial Laboratories**		**8/13/01***	
712-S	Emergency Response	pending		
713-S	**Documentation Specifications for Samples Analyzed at ECL-BERK and ECL-SC**		**4/23/03***	
714-S	Data Management of Automated Data System	pending		
715-S	**QC Rules for Organic Analyses (GC and HPLC)**		**3/17/03***	
716-S	QC Rules for Organic Analyses (GC/MS)		pending	

SOP #	Title of SOP	Status (date completed) Draft	Final	Archived
717-S	QC Rules for Inorganic Analysis		pending	
718-S	**Audit of Contract Laboratory Data**		**4/20/04***	
719-S	**ATP Review**		**2/6/03***	
720-S	**Requests for Consultation, Data Validation, Data Review and Laboratory Audits**		**4/21/04***	
721-S	**Procedures for the Distribution of Laboratory Sample Analysis Reports**		**7/10/02***	
722-S	**Procedures for Calculating Control Limits for Accuracy and Precision**		**4/20/04***	
723-S	Confirmation Procedures for Organic Methods			pending
724-S	Confirmation Procedures for Inorganic Methods			pending
725-S	**Analysis of Samples from Clandestine Drug Labs.**		**6/29/99***	
726-S	**Handling Client Feedback and Complaints**		**2/10/03***	
727-S	**Soil and Solid Waste Sampling for VOCs**			**7/18/02***
728-S	**Requesting Out of State Travel**		**10/14/98***	
729-S	**Procedures for the Handling and Extraction of Soil Samples in ENCORE Samplers for VOC Analysis**	**6/10/04***		
730-S	**Determination of % Dry Solids**		**4/19/04***	
731-S	**Toxicity Characteristic Leaching Procedure**		**5/17/04***	
732-S	**Guide for Field Soil Sampling with Encore Sampler for VOCs Analysis**	**6/10/04***		
734-S	**N-Methylcarbamates by HPLC**			**3/20/02***
735-S	Investigation of Laboratories	pending		
736-S	**Acquiring Missing Information on the SAR**	**1/21/03***		

SOP #	Title of SOP	Status (date completed) Draft	Final	Archived
737-S	Laboratory Electronic Analytical Report Management w/ LIMS	pending		
738-S	LIMS: Data backup and Recovery for Empower Chromatography Data System	pending		
739-S	**Modified EPA Method 1664: n-Hexane Extractable Material (HEM) and Silica and Sludges by Extraction and Gravimetry**	**6/10/04***		
740-S	**Determination of the Less than 100 micron Fraction of Solid Samples**		**6/10/04***	
741-S	**Request for Purchase of Equipment and Supplies**		**10/25/99***	
742-S	**Request for Purchase of Supplies with a Blanket Purchase Order**	**9/30/99***		
750-S	LIMS Work Flow of ECL Sample Tracking	pending		
760-S	**Wipe Sampling and Extraction Protocol for Pesticides and PCB's Analysis**			**5/24/04***
765-S	Analysis of Methamphetamine by GC/MS	pending		
780-S	Screening Test Method for Water Reactivity		pending	
785-S	**EDB and DBCP in Soil**			**3/13/02***
792-S	**Total Halogens in Oil**			**3/22/03***
818-S	Total Volatile Petroleum Hydrocarbons by GC/FID Headspace Analysis			9/91
820-S	Purge and Trap Method for Halogenated Volatile Organics			3/24/87
821-S	Halogenated Volatile Organics			3/24/87
830-S	**Analysis of 1,4-Dioxane in Water by Closed-System Purge-and-Trap and Gas Chromatography/Mass Spectrometry with Selected Ion Monitoring**		**6/10/04***	
850-S	GC/MS Headspace Method for Volatile Organics		pending	
855-S	Sample Preparation - Modified Method 5035	pending		
860-S	Total Organic Carbon In Soil and Sediments			5/27/94
870-S	**Procedure for Liquid Waste Handling and Disposal using Glass Bottles**		**7/1/03***	
871-S	Procedure for the Analysis of human Serum for Organochlorine Pesticides			pending
881-S	**Washing Environmental glassware for PCDD and PCDF**		**11/22/04***	

SOP #	Title of SOP	Status (date completed)		
		Draft	Final	Archived
882-S	**Washing Biological glassware for PCDD and PCDF**		**7/10/02***	
883-S	**Lipid Determination of Human Adipose Samples**		**11/22/04***	
884-S	**Extraction of Human Adipose Samples**		**11/22/04***	
885-S	**Procedure for Assembling and Operating a Disposal Carbon Column for the Analysis of PCDD/PCDFs in Environmental Samples**		**7/10/03***	
886-S	**Modified Smith/Stalling Procedure for the Analysis of PCDD/PCDFs and coplanar PCBs in Biological Samples**		**11/22/04***	
887-S	**Use of Fluid Management System's (FMS) Automated Gel Permeation Chromatography (GPC) and Florisil Column in the Clean-up of Adipose Samples for PCB and OCP Analyses**		**11/22/04***	
888-S	**Procedure for the Extraction and clean-up of Milk Samples for PCDD/PCDF, PCB, PBDE and OCP Analysis**	**7/15/01***		
889-S	**Safety Considerations When Using a Torch to Seal Ampules**		**11/22/04***	
890-S	Extraction, Clean-up and Lipid Determination of Fish Samples for PCDD/PCDF, Coplanar PCB, PBDE Analysis	11/22/04*		
891-S	Preparation and Analysis of Blood Serum for PCBs, OCPs, and PBDEs	pending		
892-S	Apparatus and Materials Used in Preparations of Samples for Trace Organohalogens	pending		
894-S	Gas tank Use & Monitoring	pending		
900-S	**Equipment Calibration Procedure for Pipets**		**9/20/05 ***	
901-S	**Equipment Calibration Procedure for Dispensers/Dilutors**		**9/21/05 ***	
902-S	**Glassware Cleaning for Metal Determinations**		**9/20/05 ***	
903-S	**Equipment Calibration Procedure for Balances**		**4/9/04 ***	
904-S	**Guidelines for Choosing the Proper Analytical Method for TPH Analysis**		**6/10/04 ***	
906-S	Digestion of Water and Liquid Samples for Metals Determination			2/10/98
907-S	Preparation of Water Samples for AAS or ICPAES Analysis			2/11/98
908-S	Digestion of Soil, Solid Waste or Sludge for Metal Determinations			6/5/90

SOP #	Title of SOP	Status (date completed)		
		Draft	Final	Archived
909-S	Digestion of Oil and Oily Samples for Metals Determinations			9/9/99
910-S	California Waste Extraction Test		6/10/03*	
911-S	Microwave Assisted Acid Digestion of Oil, and Oily Samples for Metal Determinations			2/24/98
912-S	Fluorescent Lamp Preparation for Metals, Mercury, WET and TCLP Determinations	6/10/03		
913-S	Cathode Ray Tube Preparation for Metals, Mercury, WET and TCLP Determination	6/10/03		
914-S	Preparation of Cold cathode Fluorescent Lamp Preparation for Ha Testing including WET and TOP		1/26/04*	
915-S	Toxicity Characteristic Leaching Procedure for Metals			2/9/98
916-S	Preparation of Consumer Electronic Devices Containing Liquid Crystal Displays (LCDs) for Metals, WET & TCLP		1/26/04*	
920-S	pH Electrometric Measurement			2/11/98
930-S	Determination of Metals by Flame Atomic Absorption Spectrometry			2/10/98
932-S	Determination of Metals by Graphite Furnace Atomic Absorption Spectrometry (GFAAS)			2/10/98
936-S	Determination of Hg by Cold Vapor Technique			2/10/98
940-S	**Dry Cell Battery Preparation for Metals, pH, Alkalinity, Wet and TCLP Determinations**			**4/9/04 ***
955-S	Extraction of Percholorate in Soil, Sludge and Solid Samples	pending		
960-S	Determination of Anions by Ion Chromatography			3/2/98
970-S	Headspace Analysis for Hydrofluoric Acid			12/27/91
980-S	Distillation and Screening for Cyanide			2/11/98
983-S	Determination of Cyanide by Ion-Selective Electrode			3/13/90
984-S	Reactive Cyanide			9/9/99

SOP #	Title of SOP	Status (date completed)		
		Draft	Final	Archived
991-S	Reactive Sulfide			9/9/99
992-S	Titrimetric, Iodine for Sulfide			2/11/98
993-S	Determination of Sulfide by Methylene Blue Method			2/11/98

4.2 MOBILE LABORATORY

The DTSC mobile laboratory (ML) funded by the Governor's Office of Homeland Security Grant Program was received in April of 2005. The primary objective is to respond and detect chemical releases following a natural disaster, industrial spill, or act g chemical terrorism. The secondary objectives are to support departmental projects such as site investigations, remediation and enforcement. The ML is an extension of ECL's analytical capabilities to the field to expedite clean up activities for the protection of public health and the environment.

The ML includes two separate self- contained compartments, one for sample preparations and one for sample analyses. The ML is well equipped with safety features to protect the instrument operators and the environment. The sample preparation compartment includes high efficiency particulate air filtration (HEPA) systems, a glovebox with bio-decontamination system for operator's protection, a fume hood and other common safety features. The analytical compartment is equipped with state-of-the-art measurement systems.

4.2.1 Gas chromatograph/mass spectrometers (GC/MS)

Two on-board gas chromatograph/mass spectrometers (GC/MS) are specially configured for the identification and confirmation of unknown chemicals in air, solid, and liquid samples. These include:

Agilent 6890 GC/5973 MSD with dual-wavelength Flame Photometric Detectors (FPD) is designed for detecting chemical agents, specifically compounds containing sulfur and phosphorus. A Dynatherm Model IACEM 980 Thermal Desorber is attached to the unit for air analysis. Two different GC columns are installed in this unit, one for detecting volatile organic compounds (VOC) and one for detecting toxic chemical agents. Automatic Mass Spectral Deconvolution and Identification Software (AMDIS) and retention time locking procedures are included in the operating system for automatic identification of a list of target chemical agents without depending on the presence of chemical standards.

Agilent 6850 GC/ 5973 MSD with an auto-sampler is designed for detecting toxic industrial chemicals. A ChemStation with a RTL pesticide MS Library enhances the identification and confirmation of pesticides.

4.2.2 Field portable analytical instruments

A number of field portable analytical instruments are acquired for fast on-site analysis. These include:

INFICON HAPSITE Three units of field-portable GC/MS for the detection and confirmation of VOCs in air, water, and soil. The unit can be taken to the "hot zone" by a specifically trained first respondent for on site sampling and analyses. Alternatively, they can be used to identify volatile chemicals.

X- Ray Fluorescence Analyzer (XRF) A hand-held XRF analyzer (Oxford X-Met 300TX) for screening elements in solids and liquids.

Fourier Transform Infrared Reflectometer (FTIR) A Smith Detector Travel IR HCI - HazMat Chemical Identification for organic analysis of solids and liquids

DU-Spectrometer A Beckman spectrometer for analyzing cyanide and chromium (+6) in samples

Raman Spectrometer a handheld Ahura First Defender that has the capability to directly analyze organic compounds in solids and liquids inside a container, without exposing the chemist to hazardous substances

pH Meter for waste classifications

Radiation Detectors Three units of hand – survey radioactive monitors for checking alpha, beta and gamma rays in the sample

HazMat Test Kits for quick identifications of hazardous substances to guide the laboratory analysis

Cameo/Aloha Meteorological Station A wireless weather station to monitor weather conditions for wind direction, wind speed, temperature, humility, etc., to facilitate the selection of sampling locations and for plume modeling.

Satellite System A satellite system is installed in the ML for communication and data transmission to ECL and other state and federal fixed laboratories for data sharing and decision making during an emergency response.

Method development for analyzing chemicals of concern is in progress. When the ML analytical capabilities are established and the standard operating procedures are available, the procedures for requesting the deployment of ML and field instruments will be posted in the DTSC internet: http://www.dtsc.ca.gov/AssessingRisk/HML/Mobile_Lab.cfm

Contacts

Dr. Ruth R. Chang
Senior Hazardous Substances Scientist
Department of Toxic Substances Control
Environmental Chemistry Laboratory
700 Heinz Avenue
Berkeley, CA 94710
rchang@dtsc.ca.gov

4.3 ENVIRONMENTAL CHEMISTRY LABORATORY - SOUTHERN CALIFORNIA.

4.3.1 Introduction.

The Environmental Chemistry Laboratory - Southern California (ECL-SC) is a branch of ECL and has analytical capabilities similar to ECL. The laboratory also provides method development and technical consultation services. ECL-SC has recently developed an improved method for the wet ashing of soil and sludge samples for metal analysis and methods development in the analysis for gasoline in soils. ECL-SC also supports the Pollution Prevention Program in the evaluation of new processes for the treatment and recycling of hazardous waste by recommending and providing the appropriate analytical services.

4.3.2 Analytical Requests.

See Section 4.1.2.

4.4 DHS ENVIRONMENTAL HEALTH LABORATORY (EHL) (FORMERLY THE AIR AND INDUSTRIAL HYGIENE LABORATORY).

4.4.1 Introduction.

EHL provides analytical services and leadership in the development of laboratory methods, and carries out research programs to assist State, Federal, and County agencies in the identification, evaluation, and control of public health hazards associated with airborne toxic materials. On matters pertaining to hazardous waste EHL's air vector component works very closely with the Environmental Chemistry Laboratory (ECL) especially in the area of air sampling and analysis. The work involves five interrelated functional tasks as follows:

1) Analytical Services - EHL provides analyses of air samples collected by Department of Toxic Substances Control (DTSC) and other related agencies, primarily for QC/QA purposes, and where analytical resources are unavailable on a routine basis.
2) Field Studies - EHL participates in various site characterization investigations involving air monitoring of landfill and active dump sites. EHL staff serves as technical observers and monitors in remedial and feasibility studies.

3) Training - Because of its expertise in air sampling and analysis, EHL periodically provides training to DTSC staff on basic aerometric techniques and related analytical procedures.

4) Method Development - EHL evaluates, develops, and designs effective air sampling and analytical methods crucial for the generation of high quality data for enforcement and compliance purposes. Also, staff actively participates in interlaboratory studies.

5) Consultation - EHL provides aerometric information and guidelines to DTSC personnel on matters associated with site characterization projects; critiques proposed work plan protocols and evaluates results of completed projects; participates in interagency meetings involving air monitoring strategies and recommends quality control and quality assurance measures.

4.4.2. Environmental Health Laboratory Services.

Sample Analysis:

1) ECL may allocate special analyses to EHL, or by prior arrangement DTSC units submit samples directly to EHL.

2) ECL request form, (DHS 8002) is to accompany the samples with the following further specifications: "**for EHL**".

a) Under g. field information list the total air volume collected for all air

samples.

b) Use separate forms for each type of sample i.e., air, material, or biological.

c) In listing the samples group together (or use a separate form) those requiring the same analysis, or set of analyses along with their blanks.

d) Requester's phone number, preferably ATSS, is helpful should questions arise about the request. Include a mailing address as well as the person to whom the report should be sent.

3) Should a hi-vol filter sample be submitted, use the 24-Hour Data Air Sample Report Form (TSD-2). County, site, and project codes (upper right) do not apply. Instrument number and date of last calibration are required to relate the flow meter reading to true air flow. For sampling times, the exact start and finish times should be entered and reconciled with the elapsed time meter readings, if available. The lower part of the form, pollutant description, codes and analysis are for lab use only. Only the original copy is required by the lab, and the other copies are available for the requester. Examples are available from EHL.

Sampling Consultation:

EHL has considerable expertise in air sampling and analysis. Sampling and analytical procedures are essentially interdependent functions and hence, personnel involved in aerometric field studies should consult with EHL prior to conducting the investigation. This requirement assures that air samples collected in the field are compatible with established analytical methods.

4.5 CONTRACT LABORATORIES.

The Department contracts with commercial laboratories for chemical laboratory services to supplement the services provided by ECL and ECL-SC. This contract is in addition to the contract laboratories used for the Bond Act implementation. Methods, turnaround times, and quality assurance requirements for water and solid wastes are listed in tables 4.4-1 and 4.4-2, respectively. The contract lab service is managed by ECL. When ECL is at full capacity, the excess samples will be sent to the contract lab, as directed by ECL. Sample collectors must not send samples to the contract lab without prior authorization from the ECL Sample Management Officer (SMO). Analytical and Quality Control Reports from the contract labs are sent directly to the sample collectors. A copy of all Analytical and Quality Control Reports, along with complete data packages, are sent to the contract manager at ECL. The contract manager uses this information to both ensure contract compliance and track workload status at the contract labs. The Quality Assurance and Data Validation group works closely with the contract manager to ensure that the work performed is in compliance with contract specifications. In addition, data validation can be performed on special requests for select data packages. Questions regarding data validation should be referred to Cindy Dingman at (510) 540-2329 or Lorna Garcia at (510) 540-2441.

Table 4.4-1 Water Analysis Methods and Quality Assurance Requirements.

CATEGORY	METHOD #	REFERENCE	DETECTION LIMIT (ug/L)	ACCURACY %	PRECISION[a] %	QA/QC PROTOCOL
Sb,As,Be,Ba, Cd,Cr,Co,Cu Pb,Mo,Ni,Se, Ag,Tl,V,Zn	200 Series	a	10-100[c]	80 - 120	20	A,B,C,D,E
Mercury	245	a	0.5	80 - 120	20	A,B,C,D,E
Chromium (VI)	218.5	a	50	85 - 115	10	A,B,C,D,E
Sulfide	376	a	100	85 - 115	10	A,B,C,D,E
Cyanide	335	a	40	85 - 115	10	A,B,C,D,E
Fluoride	340/300[b]	a	100	85 - 115	15	A,B,C,D,E
Chloride	325/300[b]	a	3000	85 - 115	15	A,B,C,D,E
Nitrite	354/300[b]	a	300	85 - 115	15	A,B,C,D,E
Nitrate	352/300[b]	a	300	85 - 115	15	A,B,C,D,E
Sulfate	375/300[b]	a	5000	85 - 115	15	A,B,C,D,E
Purgeable Halocarbon	601	b	0.02-2.0[c]	70 - 110[c]	25	A,B,C,D,E
Purgeable Aromatics	602	b	0.2-4.0[c]	40 - 110[c]	25	A,B,C,D,E
Phenols	604	b	0.2-20[c]	40 - 110[c]	20	A,B,C,D,E
Organochlorine Pesticides and PCBs	608	b	0.02-1.0	85 - 115	10	A,B,C,D,E
PAHs	610	b	0.02-2.5[c]	80 - 120[c]	15	A,B,C,D,E
Organophosphorus Pesticides	614/622	c	0.02-5.0[c]	50 - 120[c]	20	A,B,C,D,E
Chlorophenoxy Herbicides	509B	d	10	60 - 110[c]	15	A,B,C,D,E
Purgeables	624	b	5.0-10[c]	60 - 145[c]	25	A,B,C,D,E
CATEGORY	METHOD #	REFERENCE				

			DETECTION LIMIT (ug/L)	ACCURACY %	PRECISION[a] %	QA/QC PROTOCOL
Base/Neutral & Acids	625	b	10-50[c]	10 - 130[c]	50	A,B,C,D,E
Carbamates	632	c	0.01-0.5[c]	40 - 110[c]	15	A,B,C,D,E
pH	150	a				A,C
Fish Bioassay	Section 66696 (a)(4)	e				A,F,G

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[a] Maximum relative percent difference (RPD) of duplicates at ten or more times the limit of detection.

[b] The Determination of Inorganic Anions in Water by Ion Chromatography - Method 300.0, Test Method, EPA-600/4-84-017, March 1984.

[c] Check methods for values of specific species.

a Methods for Chemical Analysis of Water & Wastes, EPA 600/4-79-020

b "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act", 40 CFR Part 136, EPA, October 26, 1984.

c Nonconventional Pesticides Chemicals Analysis of Industrial and Municipal Wastewater, Test Methods, EPA-440/1-83/079-C.

d Standard Methods for the Examination of Water and Wastewater, 16th Edition, 1985.

e California Administrative Code, Title 22, Chapter 30, Article 11, "Criteria for Identification of Hazardous and Extremely Hazardous Wastes".

QA/QC PROTOCOLS:

A All QA/QC procedures required by the method.

B One method blank for every ten samples or batch of samples or type of matrix, whichever is more frequent.

C One sample analyzed in duplicate for every ten samples or batch of samples or type of matrix, whichever is more frequent.

D One spiked sample for every ten samples or batch of samples or type of matrix whichever is more frequent. Spike shall be made at ten times the detection limit or at the analyte level.

E Analyze quality control sample (if available) with every ten samples or batch of samples or type of matrix, whichever is more frequent.

F One control for each sample.

G Bioassay screening at 250 mg/L and 750 mg/L, at a minimum.

Table 4.4-2 Soil, Liquid Waste, and Solid Waste Analysis Methods, and Quality Assurance Requirements.

CATEGORY	METHOD #	REFERENCE	DETECTION LIMIT mg/Kg	ACCURACY %	PRECISION[a] %	QA/QC PROTOCOL
Sb,As,Be,Ba, Cd,Cr,Co,Cu Pb,Mo,Ni,Se, Ag,Tl,V,Zn	Section 66700	a	1.0	75 - 125	35	A,B,C,D,E
Mercury (Hg)	7471B	a	1.0	75 - 125	35	A,B,C,D,E
Chromium (VI)	7195/6A/7	a	0.5	80 - 120	35	A,B,C,D,E
Sulfide	9030B	a	10	80 - 120	15	A,B,C,D,E
Cyanide	9010B	a	5	80 - 120	15	A,B,C,D,E
Fluoride	340/300	b,c	10	80 - 120	20	A,B,C,D,E
Chloride	325/300	b,c	100	80 - 120	20	A,B,C,D,E
Nitrite	354/300	b,c	10	80 - 120	20	A,B,C,D,E
Nitrate	352/300	b,c	10	80 - 120	20	A,B,C,D,E
Sulfate	375/300	b,c	100	80 - 120	20	A,B,C,D,E
Waste Extraction Test (WET)[c]	Section 66700	d	0.1 mg/L	75 - 125	35	A,B,C
Halogenated Volatile Organics	8010B	a	0.2-20[d]	30 - 110[d]	50	A,B,C,D,E
Aromatic Volatile Organics	8021B	a	2.0-40[d]	30 - 110[d]	50	A,B,C,D,E
Phenols	8041	a	0.2-20[d]	30 - 140[d]	40	A,B,C,D,E
Organochlorine Pesticides	8081A	a	0.5-10[d]	25 - 140	25	A,B,C,D,E
PCBs	8082	a	0.5-10[d]	25 - 140	25	A,B,C,D,E
PAHs	8100/8310	a	0.2-20[d]	50 - 120[d]	25	A,B,C,D,E
Organophosphorus Pesticides	8141A	a	1.0-20[d]	50 - 120[d]	25	A,B,C,D,E

CATEGORY	METHOD #	REFERENCE	DETECTION	ACCURACY	PRECISION[a]	QA/QC

			LIMIT mg/Kg	%	%	PROTOCOL
Chlorophenoxy Herbicides	8151A	a	1	50 - 110[d]	20	A,B,C,D,E
GC/MS Method: Volatile Organics	8260B	a	1	60 - 140[d]	20	A,B,C,D,E
GC/MS Method: Semi-Volatile Organics	8270C	a	1-5[d]	30 - 140[d]	20	A,B,C,D,E
pH	9040B	a				A
Fish Bioassay	Section 66696 (a)(4)	d				A,F,G
Ignitability	1010/1020	a				A
Corrosivity[e]	Section 66708	d				A
Reactivity[d]	Section 66705	d				A

[a] Maximum relative percent difference (RPD) of duplicates, at ten or more times the limit of detection.

[b] Method may be modified to use specific ion electrode or colorimetry.

[c] Extraction and analysis of antimony, arsenic, beryllium, barium, cadmium, chromium, cobalt, copper, lead, molybdenum, nickel, selenium, silver, thallium, vanadium, zinc, and mercury.

[d] Check methods for values of specific species.

[e] Test by corrosivity toward steel.

[f] Water reactivity and cyanide and sulfide screening.

a Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW- 846, 2nd Edition, U. S. EPA, revised April 1984, or 3rd Edition, 1986.

b Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020.

c The Determination of Inorganic Anions in Water by Ion Chromatography - Method 300.0, Test Method, EPA-600/4-84-017, March 1984. Sample preparation such as aqueous extractions may be needed.

d California Administrative Code, Title 22, Chapter 30, Article 11, "Criteria for Identification of Hazardous and Extremely Hazardous Wastes".

QA/QC PROTOCOLS:

A All QA/QC procedures required by the methods.

B One method blank for every ten samples or batch of samples or type of matrix, whichever is more frequent.

C One sample analyzed in duplicate for every ten samples or batch of samples or type of matrix, whichever is more frequent.

D One spiked sample for every ten samples or batch of samples or type of matrix, whichever is more frequent. Spikes shall be made at ten times the detection limit or at the analyte level.

E Analyze EPA quality control sample (if available) and/or NBS traceable standards (if available) with every ten samples or batch of samples or type of matrix, whichever is more frequent.

F One control for each sample.

G Bioassay screening at 250 mg/L and 750 mg/L, at a minimum.

4.6 LABORATORY ACCREDITATION.

The California Hazardous Waste Laboratory Certification Program was created in 1982 (AB 3449, Chapter 1209, Statutes of 1982). The bill directed the Department to adopt regulations governing the criteria for certification, the certification process, and the procedures to be used by hazardous waste laboratories to analyze and identify waste samples. The certification regulations were adopted on April 12, 1985. Effective April 11, 1986, any analysis of hazardous waste required by the Hazardous Waste Control Law must be performed by certified laboratories. Minimum requirements were established for:

Test procedures
Quality assurance programs
Laboratory equipment
Personnel qualifications

Mechanisms were prescribed to monitor the operation and performance of a laboratory through site inspections and proficiency testing.

During 1988, Assembly Bill 3739 (Jones) was signed into law. The law was amended by AB 2160 (Bronzan) in 1989 and AB 45 (Jones) in 1990. This law consolidated the Hazardous Waste Testing Laboratory Certification program with an existing Water Testing Laboratory Certification program, a pesticide testing in food program and a new wastewater testing accreditation program. Certification regulations have been adopted by the DHS Environmental Laboratory Accreditation Program (ELAP) to certify/accredit environmental testing laboratories. SB 1304 (Committee on Environmental Quality, Senator Sher, Chair), 1999; and SB 2203 (Committee on Environmental Quality, Senator Sher, Chair), 2000 authorized ELAP to participate in a national environmental laboratory accreditation program according to the standards adopted by the National Environmental Laboratory Accreditation Conference (NELAC). This national program is administered by the Director of the National Environmental Laboratory Accreditation Program (NELAP). California is a recognized Accrediting Authority to grant NELAP accreditation to an applicant

laboratory. Thus, an environmental laboratory needs to be either certified under ELAP or accredited under NELAP in order to perform environmental testing for regulatory purposes

Questions regarding lab accreditation should be directed to ELAP at (510) 540-2800.

Internet address:

ELAP http://www.dhs.ca.gov/ps/ls/ELAP/default.htm

NELAC http:/www.epa.gov/ordntrnt/ORD/nelac/index.html

5.0. QUALITY CONTROL

The goal of a quality assurance program is to produce data that are of known and acceptable quality. The quality of the data depends on procedures followed both in the field and in the laboratory. Therefore, part of the quality assurance program is the collection and analysis of quality control samples.

5.1. SAMPLING QUALITY CONTROL.

The sampling plan should include specific quality control requirements to meet the data quality objectives. These objectives are usually met through the use of quality control samples such as replicates, blanks, and sometimes performance evaluation samples. Unless the specific data needs at a site allow lesser or stricter quality assurance, the minimum guidelines given in the following sections apply:

5.1.1 Quality Control Samples for Sampling.

Replicates should be collected at a minimum frequency of 5%, with minimum of one set of duplicates per batch. Travel blanks should be submitted with each sample shipment. Field and equipment blanks, if needed, should be collected at the frequency of one per sampling day.

Field replicate samples:

Field replicate samples consist of either co-located samples (i.e., samples collected consecutively from nearly the same location) or split samples (i.e., samples which have been divided up from one homogenized sample). Co-located samples allow an estimate to be made of total variability, including sampling variability, whereas split samples are better for estimating transportation and laboratory variability. Replicates should be collected at a minimum rate of 5%, that is, one well in every 20 wells should be sampled twice for co-located samples or twice as much water collected, homogenized, and split into twice as many containers for split samples. Split samples are also used when two different parties are sampling the same site and verification of analytical results is necessary.

Sampling blanks:

Blank samples should be included in order to determine if field procedures such as collection, preservation, shipping, or decontamination are resulting in contamination of the samples, thereby yielding concentrations of some parameters of interest higher than actually exist in the field. These blank samples are of several kinds:

- Equipment blanks are collected when sampling equipment is decontaminated in the field as a check on the decontamination procedure. Equipment blanks may be relevant whenever the integrity of the sampling equipment is questioned. For water samples, clean water is collected using the equipment in question and sent to the lab with the other samples for analysis. In some cases, if holding times are not too short, the equipment blanks may be collected and stored and analyzed only if contaminants are found in the samples.

- Travel blanks (or trip blanks) should accompany sample containers to and from the field. They consist of sample containers which are filled in the laboratory with purified water, taken into the field, and added to each cooler before it is transported to the lab. These travel blanks are especially important for volatile samples in which other samples containing high concentration of parameters of interest may leak in the cooler and contaminate other samples. Travel blanks are generally not used for other media such as soil.

- Field blanks should be collected at specified frequencies, which will vary according to the probability of contamination or cross-contamination. They consist of purified water, such as HPLC Grade or pesticide grade water (in the case of water samples) which is taken into the field and transferred from the water container to the individual sample containers in the field as a check on contamination in the atmosphere at the site. The purpose of the field blank is to verify that none of the analytes of interest measured in the field samples resulted from contamination of the samples during sampling. If the purified water is also poured through sampling equipment before being added to the sample containers, the field blanks may also substitute for equipment blanks.

- Temperature blanks, appropriate contained filled with water should be submitted for each cooler.

Background samples:
Background samples are collected from the site in the exact manner as the regular samples. Background samples may be used as a quality control sample, especially for non-aqueous samples, to look for sampling or laboratory effects on concentration. The more common use for background samples is to establish a background concentration for those parameters which occur in the area of the site.

Control and/or spiked samples:

- Spiked or control samples: Performance evaluation (PE) samples include all spiked samples and standard solutions of known composition included with the samples sent to the laboratory from the field as a measure of the potential loss of analyte on

shipping and for recovery of analytes from a particular medium. Field spikes may also be desired when preservation techniques are in question. These include field samples which are spiked with a known amount of a parameter(s). They also include standard solutions of a known concentration obtained from a laboratory (e.g., ECL or EPA laboratory) or commercial source, labeled as a sample, and sent blind to the laboratory.

5.1.2 Minimum Recommended for Sampling QC Samples.

SAMPLE TYPE	RATE, BASED ON TOTAL SAMPLE LOAD
Replicate Samples:	
• Co-located or split	5% or 1 per sampling event for each type of media or location sampled.
Blanks:	
• Travel Blanks[1]	1 per sample for each procedure, for each sampling event.
• Temperature Blanks[1]	1 per sample for each cooler.
• Equipment Blanks[1]	1 per decontamination event in the field (as needed).
Spike or Control Samples[1]	5% for each procedure if the integrity of the sample warrants it.
Background Samples - may be used for matrix spiking	Varies, see Section 5.1

[1] May not be necessary possible with all sample types

5.1.3 QC Samples for a Sampling Activity.

Assume a sampling project consists of six water samples and three soil samples over a period of two days. The analyses required are volatile organics (EPA SW 846 Method 8260), metals, plus semi-volatile organics (EPA SW 846 Method 8270). The total number of samples collected would be:

Day 1:

Equipment Blank (if needed to confirm decontamination):
1 x 1L (8270), 1 x 1L (metals), 2 x 40mL (8260)

Well 1: 1 x 1L (8270), 1 x 1L (metals), 2 x 40mL (8260)

Well 2: 1 x 1L (8270), 1 x 1L (metals), 2 x 40mL (8240)

Well 3: 1 x 1L (8270), 1 x 1L (metals), 2 x 40mL (8260)

Well 4: 1 x 1L (8270), 1 x 1L (metals), 2 x 40mL (8260)

Well 4 Replicate (split): 1 x 1L (8270), 1 x 1L (metals), 2 x 40mL (8260)

Travel Blank: 2 x 40mL (8260), 1 x 1L (8270), 1 x 1L (metals)

Day 2:

Equipment Blank (if needed to confirm decontamination):
1 x 1L (8270), 1 x 1L (metals), 2 x 40mL (8260)

Well 5: 1 x 1L (8270), 1 x 1L (metals), 2 x 40mL (8260)

Well 6: 1 x 1L (8270), 1 x 1L (metals), 2 x 40mL (8260)

Travel Blank: 2 x 40mL (VOA)

Soil 1:1 x 200g.(8270, metals),1 Single transfer sampler e.g. Encore™ (8260)

Soil 2:1 x 200g.(8270, metals),1 Single transfer sampler e.g. Encore™ (8260)

Soil 3: 1x200g.(8270, metals),1 Single transfer sampler e.g. Encore™ (8260)

Soil 3 duplicate: 1 x 200g.(8270, metals),1 Single transfer sampler e.g. Encore™ (8260)

Control Sample: 1 x 100g. (if available, for 8270)

Control Sample: 1 x 200g. (if available, for metals)

5.2 LABORATORY QUALITY CONTROL

5.2.1 Routine Laboratory Quality Control Practices.

Laboratory blanks, and spike samples are usually analyzed at a minimum of once for every batch of samples or type of matrix or 20 samples, whichever is more frequent.

Duplicate samples are analyzed at a minimum of once for every batch of samples or type of matrix or 20 samples, which ever is more frequent.

The spike contains target analytes, typically spiked either at the level of each analyte present or at the concentration of the mid-range calibration standard, whichever is higher. If it is of significance to know the accuracy at the regulatory limit, spikes may be made at this regulatory limit.

The following general quality control practices are employed at ECL on a routine basis:

- At least a three point calibration for inorganic analysis (except ICP) and a 5 point calibration for organic analysis are performed on all sample analysis. If a calibration blank is analyzed, this will constitute an additional point.

- A calibration standard is analyzed to verify the calibration. This method standard consists of a reference standard from a different source than the calibration standards. The concentration of this standard is near the mid-point of the calibration curve.

- A method blank is analyzed to demonstrate the existence, if any, of contamination and its magnitude.

- Duplicates are analyzed to measure subsampling and analytical precision. At times, a matrix spike (MS) and a matrix spike duplicate (MSD) are used for this measure instead of the duplicates.

- A spiked sample is analyzed to measure accuracy (or bias) in sample preparation and analysis.

In addition, the following quality control samples may be included for special projects:

- Internal quality control samples of known composition. These samples may be submitted as blind samples to the laboratory.

Further, specialized analytical techniques such as GC/MS and ICP may have additional

quality control requirements specified by the analytical methods. These include tuning data for GC/MS, interference check standards for ICP, etc.

5.3 REFERENCES.

1) Taylor, J. K. and Stanley, T. W. Quality Assurance for Environmental Measurements. ASTM STP 876. ASTM (1985).

6.0 INTERPRETATION OF ANALYTICAL DATA

The first step in the interpretation of analytical data is the review of data submitted by laboratories, responsible parties and/or project managers. During this process, the evaluator initially establishes the data validation validity.

Analytical data that exceed regulatory criteria often reflect violations of waste regulations and trigger remediation. At times, the process of remediation may involve litigation. So, analytical data can be an important component of evidence and as such should be legally defensible. It is then essential that evaluators establish validity of data to be presented as reliable evidence.

Data validation should take into account both sampling and analysis because both may contribute to errors in the results. It is important to locate sources of error arising from sampling and analysis. Merely evaluating the laboratory analysis is not a substitute for evaluating the entire process. Sampling in the field and subsequent sub-sampling in the laboratory are typically the areas where the process is the least certain. It is therefore particularly important that these steps be included when evaluating data.

6.1 GENERAL REQUIREMENTS.

Generally, Sampling and Analysis Plans (SAPs), Waste Analysis Plans (WAPs) and Quality Assurance Project Plans (QAPPs) require prior approval by EPA or Cal-EPA. Data that have been generated without the use of approved plans may have deficiencies and may be unusable for the intended purpose. On request, personnel at ECL evaluate such plans for compliance and completeness.

Field sampling procedures, laboratory analytical methods and quality assurance protocols must be clearly stated in the above documents. Laboratories performing the specified analyses must be certified/accredited by the Environmental Laboratory Accreditation Program (ELAP).

6.2 INDICATORS OF DATA QUALITY.

The typical indicators of data quality are precision which measures random error, accuracy which measures systematic error, comparability, representativeness, completeness and existence or non-existence of sample contamination. Other factors to consider are detection limits, blunders, and fraud.

6.2.1 Precision:

Precision is measurement of random error. It is the degree of agreement between two or more measurements. The simplest way to report precision is as Relative Percent

Difference (RPD). RPD is calculated as the difference between two measurements divided by their mean. The Range (R), Standard Deviation (s), and Coefficient of Variation (CV) or Relative Standard Deviation (RSD), are also used as measures of precision. A small RSD or RPD indicates high precision. RSD and RPD are fractions of the measurement which can be converted to limits about the mean. For example: 50 mg/kg ±5 mg/kg or 50 mg/kg ±10%.

Precision as relative percent difference (RPD) is calculated as:

$$RPD = \frac{|X_1 - X_2|}{\bar{x}} \times 100$$

where X_1 and X_2 are duplicate analyses and $\bar{x}$ is the mean value of the two values.

$$\%RSD = \frac{s}{\bar{x}} \times 100$$

The relative standard deviation or coefficient of variation:

where *s* is the sample standard deviation.

$$RPD = \%RSD \times \sqrt{2}$$

The relationship between the *RPD* and the *% RSD* is:

Precision for the entire measurement process is best determined using homogeneous split samples. Laboratory replicates (typically duplicates) indicate intralaboratory (within laboratory) precision for the analysis. Laboratories sometimes run duplicates through only a portion of the analysis. It is important to distinguish between those duplicates that represent the entire analysis and those that may, for example, only represent the instrumental step and exclude the sample preparation part.

Sometimes matrix spike and matrix spike duplicates (MS/MSDs) are used instead of duplicates to measure precision. When duplicates do not have any analytes, analyses of duplicates do not provide any data on precision. Analysis of MS/MSD assures the

availability of precision data.

MS and MSDs are analyzed once for every batch of samples or every matrix or every twenty samples whichever is more frequent.

Precision is determined by duplicate or MS/MSD analyses. Precision should be monitored and documented for each parameter and matrix routinely run in the laboratory. At a minimum, water and soil results should be monitored. When the control limits for precision are exceeded, corrective action should be initiated or an explanation should be included in the laboratory report.

6.2.2 Accuracy:

Accuracy (or freedom from bias) is the agreement with the true value. Accuracy is usually determined by spiking samples with a known amount of analyte. The ratio of the measured amount to known amount is termed the recovery, which is expressed as a percentage. Spiked sample accuracy as percent recovery (R) is calculated as:

$$R = \frac{(C - X)}{T} \times 100$$

where C is the measured spike sample value
X is the unspiked sample value
T is the value of spike added

The spikes are usually added to the sample before it is extracted or digested and carried through the entire preparative and analytical scheme. Such spikes are called laboratory matrix spikes. When evaluating laboratory matrix spikes, it is important to determine the point in the procedure at which the sample was spiked. A sample that was spiked before preparation is used to determine percent recovery for the entire procedure. A spike introduced later in the analysis can be used for other reasons but cannot be used to determine percent recovery for the sample preparation or extraction.

Method spikes (method blanks spiked with reference standards) are used to demonstrate that the analytical system is operating within control limits and also at times to document unusual recoveries due to matrix effects.

In addition to matrix spikes, reference materials (i.e. materials certified by NIST which contain analytes of interest at known values) can also be used to assess accuracy. Such reference materials are usually used to validate methods, evaluate laboratories and/or analysts. They may also be used as external quality control samples.

When multiple analyses of a reference sample or multiple spikes of a matrix are run, the

standard deviation of the recoveries can be calculated. While it is not common that a project would require this level of QC, it is then possible to calculate the expected accuracy with a certain confidence. Refer to Reference 1 for an example and more information.

MSs are analyzed once for every batch of samples or every matrix or every twenty samples whichever is more frequent.

Accuracy is determined by external and/or internal check samples and matrix spikes. Accuracy is monitored and documented for each parameter and matrix routinely run in the laboratory. At a minimum, water and soil matrices are monitored. When the control limits for accuracy is exceeded, corrective action must be initiated before the analysis is completed.

6.2.3 Blank analysis to measure contamination:

Assessment of blank data is an important part of data validation. Several types of blanks are used.

Field blanks are valuable because they incorporate the entire measurement process. They are prepared in the field by the sample collector. When properly designed, they are submitted blind and analyzed and reported like any other sample. Contamination may occur in the field at the time of sampling through the use of contaminated equipment. Equipment blanks are used to measure such contamination. Travel blanks are used to monitor contamination that may occur during transportation. Finally laboratory contamination is monitored by the use of method blanks. Organic solvents used in sample extraction or equipment cleaning are typical contaminants. Methylene chloride and acetone commonly appear in volatile organics results (Method 8260). Phthalates, such as bis (ethylhexyl) phthalate and di (n-octyl) phthalate, are used as plasticizers and are frequently found in semivolatile organics (Method 8270) results.

When comparing blank and sample data, consideration must be given to the dilutions used in the analysis. If a high level sample needs to be diluted for analysis, that dilution must also be made to the laboratory blank. The concentration of a parameter in the blank would then be multiplied by the dilution factor. This can be a problem in volatile organic analyses because trace level contamination in the diluent water can be interpreted as a large amount of analyte in the original sample.

Field blanks, equipment blanks, trip blanks, etc. will be analyzed in addition as submitted. Method blanks shall be preformed at one per batch of samples per matrix type per sample extraction or preparation method.

Calibration blanks which are used to set the instrument as zero response are analyzed as specified by the manufacturer and/or protocol to demonstrate that the instruments are

properly calibrated.

Method blanks are checked for any contamination problems. Routinely, they contain no analytes. A possible exception to this rule is when common laboratory solvents such as Acetone, Methylene Chloride are detected in volatile analyses. Reports may be released for a particular study pending the correction of this problem if the results of the analyses are not compromised by this contamination problem.

Although analytical data are not usually corrected for contamination in sample blanks, it should be so noted. The concentration of any analytes found in the blanks will be reported as found. Routinely blank concentrations are not subtracted from the sample concentrations. It is advised that if blank concentrations need to be subtracted from sample concentrations control charts be maintained so that the long term contamination of a laboratory is definitively established and an averaged value is subtracted. Generally, analyte concentration in blanks are of concern when they are greater than 10% of the sample concentration or the method detection limit.

6.2.4 References

1. USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review, USEPA Office of Solid Waste and Emergency Response, OLMO 4.2 May 1999

2. USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review,USEPA Office of Solid Waste and Emergency Response, ILMO 5.3 March 2004

6.3 Method Detection Limits and Quantitation Limits:

There are several conventions for reporting results near the reporting limit. When in doubt about the meaning of a result, the reporting lab should be contacted. Statistical analysis of results below the reporting limit can be done by 1) substituting a value for the non-detect result, 2) assuming a distribution from the results above the reporting limit, or 3) "robust" statistical techniques. These techniques are reviewed and discussed in Helsel, 1990 (see references).

6.3.1 Method Detection Limit (MDL)

For all methods used at ECL and ECL-SC, except trace analysis, MDL is based on the statistical calculations of replicate sample matrix spikes as defined in "Appendix B to Part 136 - Definition and Procedure for the Determination of the Method Detection Limit - Revision 1.11," CFR, 49, No. 209, Friday, 10/26/84, 198-199). Specifically, the MDL is defined as

$$\mathbf{MDL = t_{(n-1,1-\alpha=0.99)} \times s}$$

where:

$t_{(n-1,1-\alpha=0.99)}$ = the student's t value appropriate for a 99% confidence level and a standard deviation estimate with n-1 degrees of freedom.

s = the standard deviation of the replicates analyses.

For the Dioxin and Furan analysis, the MDL is defined as

$$\mathbf{MDL = 3 \times s \times F}$$

where:

s = the standard deviation of the background noise level of the actual sample extract.

F = the sample extract dilution factor times any additional factors to account for matrix interferences.

MDL is three times the noise (background) of the sample.

In both cases, the MDL is calculated for the original sample matrix and not its resultant extract or digestate, i.e., dilution factors are applied to correct extract or digestate concentration back to the original sample concentration. MDLs will be applied primarily to laboratory reagent water and clean soil and generally not to the more complicated matrices,

such as sludges, as these matrices are more difficult to define.

6.3.2 Quantitation Limit (QL)

For the Organic, GC/MS and Inorganic Units, the QL is defined as

QL = LS x F

where:

LS = the lowest acceptable calibration standard (acceptable as defined for a linear response or by actual curve fitting).

F = the sample extract dilution factor times any additional factors to account for matrix interferences.

For the Dioxin and Furan analysis, the QL is defined as

QL = 10 x s x F

where:

s = the standard deviation of the background noise level of the actual sample extract.

F = the sample extract dilution factor times any additional factors to account for matrix interferences.

6.4 Reporting Criteria

6.4.1 Tentative Identification of Non-target Sample Compounds by GC/MS analysis

For samples containing compounds not associated with the calibration standards, a computer library search may be made for the purpose of tentative identification. The reference library used for the search is the NIST/EPA MSDS mass spectral library. The necessity to perform tentative identification will be determined by the analysis objective. The identification of the compound is dependent on the chromatographic resolution and spectral quality of the unknown compound. Guidelines presented in the method are used to making tentative identifications. An estimate of the concentration for non-target compounds of the sample will be based on the total ion chromatogram (TIC) areas of the closest internal standard and non-target compound as described in the method.

6.4.2 Terms and symbols used in the Dioxin and Furan Reports

* This symbol indicates that an analyte is below the MDL (minimum detectable level). In the case where a real dioxin was detected and quantified in a sample the MDL for that isomer in the sample is based on three times the noise (background) of the average blank. The MDL for that analyte is reported.

B This symbol indicates that an analyte was detected above the MDL but below the QL. The measured value is reported.

When an analyte is above the QL, that value is reported without any accompanying symbol.

In the case where analyte is detected in both the blank and the sample:

L This symbol indicates that analyte detected in the sample is also detected in the blank. The amount in the sample is less than three times the amount in the blank. The value reported is the upper limit of the concentration that could be in the sample. The blank is not subtracted.

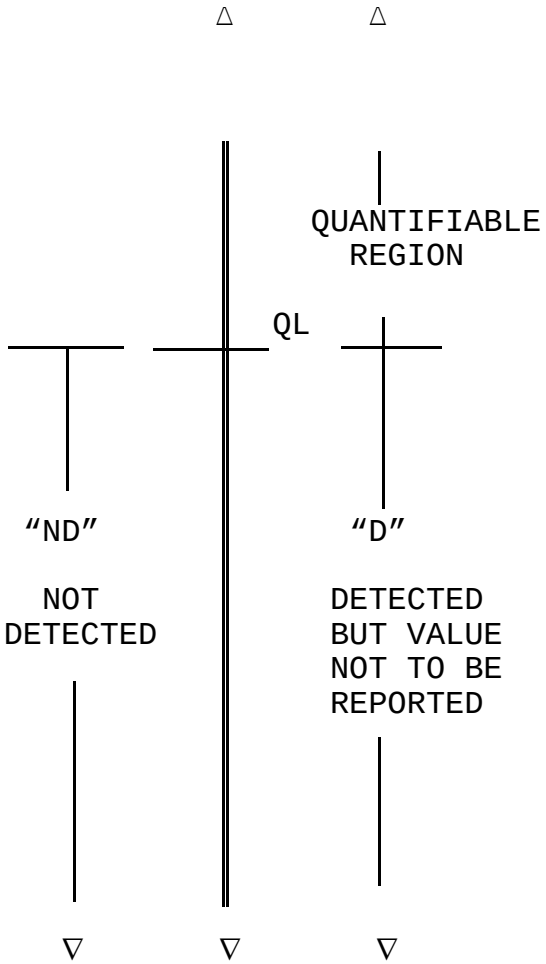
\# This symbol indicates that the analyte was detected in the blank and the sample. The amount in the sample is between three and ten times the amount detected in the blank. The value reported is the upper limit of the concentration that could be in the sample.

I This symbol indicates that the analytes was detected but interferences are present in the quantitation ion or the confirmation ion. The value reflects the upper limit of the concentration that could be in the sample.

6.4.1 Based on the previously defined limits (MDL and QL), the following diagrams describe the criteria used in ECL in the reporting of routine analytical results.
MDL not yet established (not applicable to EPA 6010 results).
Report analytical results ≥ QL.

Report detected results < QL as "D" (meaning "detected, but not quantitated, i.e. < QL), instead of the numerical results.

Report not detected results as "ND".



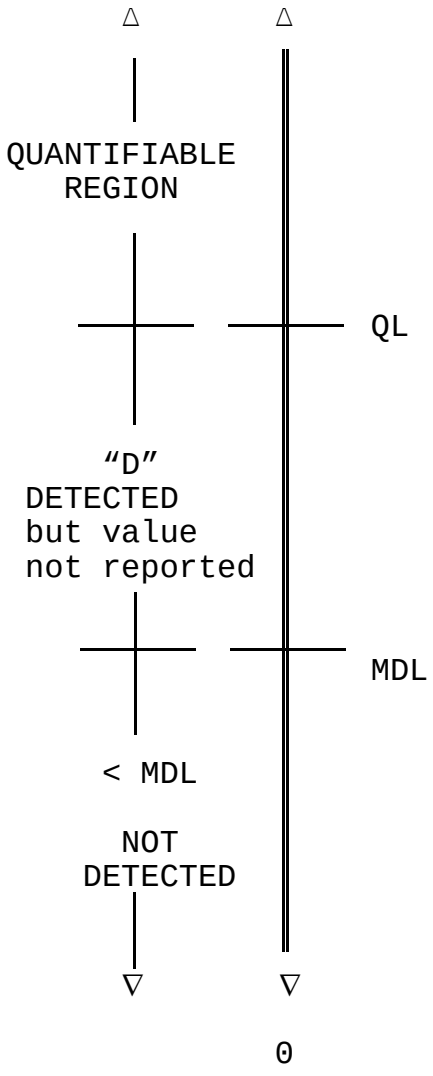
ANALYTE NOT DETECTED **ANALYTE DETECTED**

6.4.2 MDL established (not applicable to EPA 6010 results).

Report analytical results ≥ QL.

* Report results ≥ MDL but < QL, as "D" (meaning "Detected", but not quantitated I. e. < QL), instead of the numerical results.

Report results < MDL as "ND" (not detected).

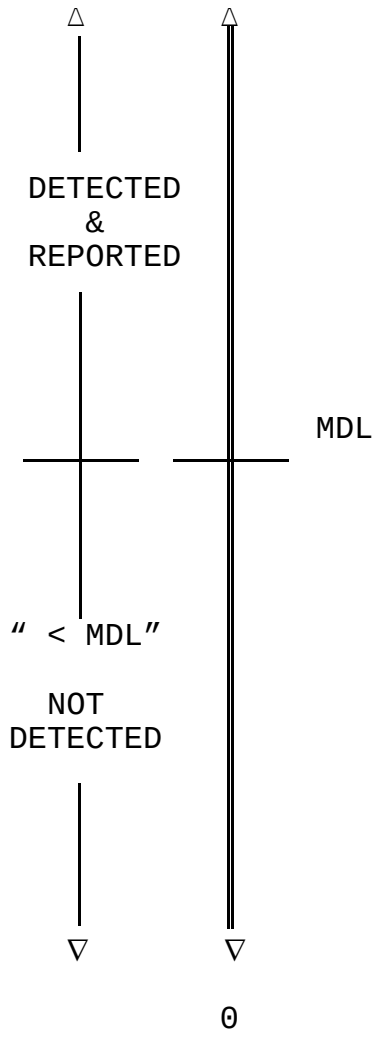


* Please note:
If requested for nonroutine analysis, estimated numerical values can be reported for results ≥ MDL but < QL

6.4.3 Reporting criteria for Method 6010 results.

Report analytical results ≥ MDL.

Report results < MDL as "< MDL value" for the respective elements.



6.5 Comparability:

In data assessment, the term comparability is used in different ways depending on the context.

It is not unusual for data from different laboratories to seemingly conflict. Typically the largest area of uncertainty is the sample itself. It is important to determine if the samples received by each laboratory were actually similar. Were the samples actual split samples? Were they shipped and preserved in the same way? Were they subsampled in the laboratory the same way? Were the samples homogenized during initial sample preparation? If the samples were not the same or if the samples were treated differently it is not reasonable to expect analytical results to agree.

Method comparison - In many cases the same method may be cited by both laboratories (e.g. EPA method 601) but the actual procedures performed may be substantially different. It is important to determine if the procedures used are appropriate to the analysis. Further, for example, if one data set were generated after an initial filtration step, the other data also should have been generated after a filtration step. If this fact cannot be documented the comparability of the data sets is in doubt.

Another common technique is the comparison of results for samples split between two or more laboratories.

Quality Control Data - Quality control sample data should be examined for all the data quality indicators given in sections 6.2. Detection limits should be compared. One laboratory may report a compound at a level of 4 mg/kg and while another laboratory reports Not Detected (ND). This is not a conflict if the limit of detection adopted by the latter laboratory is greater than 4 mg/kg. Another important consideration is blank analysis. Method blanks should be examined any time that one laboratory reports positive results and another ND, as the positive results may actually be laboratory contamination.

Ideally, interlaboratory comparisons of data should be performed only on data generated concurrently by the analysis of split samples.

One rule of thumb for labs using similar methods on homogeneous samples, is that results should agree within a factor of two (2). For example, if one lab reports 50 ppm, the other lab's results should be within 25 to 100 ppm. These limits are used by the EPA Office of Solid Waste when evaluating interlaboratory method performance data to accepted values. Where differences between labs cannot be resolved, the labs may exchange sample extracts, analyze performance evaluation samples, or re-analyze samples which are at issue. ECL can arrange these tasks for Cal-EPA staff.

6.6 Representativeness:

Representativeness refers to the extent to which the analytical results reported represents the site actually sampled. There are two areas in the sampling and analytical process that affect representativeness.

Sampling at the site by the sample collector is a crucial step in the entire sampling process in that the sample(s) collected should be representative of the material that is being sampled. If a bias is built into the sampling process, the representativeness of the sampling will be in question. Statistically valid random sampling processes should be used when sampling. If a biased sampling procedure is adopted for a particular site, adequate reasons for use of such a sampling scheme should be provided. All aspects of sampling should be well documented. Section 3 addresses these concerns in sufficient detail.

Samples brought into the laboratory are sub-sampled by the analyst to carry out a particular analytical method. This is referred to as representativeness at sub-sampling. Adequate controls must be established to assure that there is no bias introduced at subsampling. If the entire sample brought into the laboratory is homogenized and subsampled thereafter, variability due to subsampling is negligible.

6.7 Completeness:

The traditional definition of Completeness is an attempt to establish the degree of completion of the work specified in a project plan. The criterion for completion is typically set at 90%.

For example, if 5 surface samples, 10 subsurface samples and 5 well samples were collected and if each of these samples were to be analyzed for metals, VOAs, and semi-volatile organics; a project report would contain at least 90 % of the data specified. That is, a total of 20 samples with each sample analyzed by three methods, or 60 test results, should be reported. Further, each of these tests would have several analytes. Additionally, QC data specified in a QAPP would also be included in a typical report.

A more challenging aspect of completeness is to account for complete mass balance. That is, one would try to correlate the indicator parameters such as specific conductivity, total organic carbon, total organic halides, etc. to all tested analyte concentrations. If a discrepancy exists, this will give a measure of incompleteness. This approach is taken in certain specific circumstances when accounting for total mass is critical in the assessment of data quality.

6.8 Method References:

Field and analytical methods used should always be considered when evaluating data. Most methods will have QC requirements built into the procedures. Any additional QC procedures and their acceptance limits should be specified in the project plans.

Laboratory reports should include method references, sample matrix, method detection limits (MDL)/quantitation limits (QL) and reporting units. It is important that the methods used are those specified in the quality assurance project plans and are appropriate for the objectives of the study. A comparison should be made of the QC requirements in the stated method and those reported. If fewer QC samples were run than required or if QC samples reported are not within limits, then the results may not be valid. Obviously, sample data reported without method reference or QC data are highly uncertain and may not be usable for all purposes.

6.9 COMPARISON WITH REGULATORY LIMITS.

When analytical results are compared with regulatory limits, consideration must be made to recovery and precision data. In some cases, if a compound is reported as 4800 mg/L ± 10% and the regulatory limit is 5000 mg/L, then the result may be considered to be greater than the regulatory limit. This is because the confidence interval is 4320 to 5280 mg/L and the upper limit is greater than 5000. SW-846 uses the 80% confidence interval (two tailed) to determine whether a waste is hazardous. Where no uncertainty data are available and results are reported close to regulatory limits (i.e. within a factor of two), the reviewer should rely on precision data and experience with data from similar situations to determine if more analytical work is necessary.
Recovery data can play a similar role in evaluating data quality. When matrix spike samples have recoveries that are below the acceptable limits and the uncorrected results are close to regulatory limits, there may be cause for additional analysis before it can be determined that samples are actually below regulatory limits.

6.10 USABILITY OF DATA AS EVIDENTIARY MATERIAL FOR LITIGATION.

What is acceptable as legal evidence is beyond the scope of this manual. A court will usually accept evidence that is generated by methods specified by laws or generally accepted by the scientific community. For this reason laboratory and field procedures should be based on EPA, or other standard-setting organizations. Secondly, and just as important, there must be documentation to support all reported results. The correct procedures may have been used and valid data may have been generated, but they will be of limited value as evidence unless proper documentation such as chain of custody exists.

To assess data on this level, much more information is necessary than that usually contained in a laboratory report. However, this information should be available in the raw data packages for review if needed.

Considering that legal action sometimes occurs years after the fact, it is impossible for a chemist or sampler to recall from memory or even from notes every detail of a sampling or analysis. For this reason, standard operating procedures are used to ensure that operations will be consistent and analytical results will be retrievable.

6.11 DATA INTERPRETATION EXAMPLE.

The following is an example of data interpretation including both field and laboratory work. The original data package is too voluminous to include in this manual but the reader can see that many indicators of data quality are identified and discussed.

CALIFORNIA DEPARTMENT OF TOXIC SUBSTANCES CONTROL
Environmental Chemistry Laboratory

Casmalia Resources, Groundwater
Sampling Dates: 5/19/86 to 5/21/86

DATA EVALUATION

The following discussion focuses on positive laboratory results, by category of analysis. The superscripts (i.e., [a], [b], and [c]) denote the three laboratories conducting the analyses. For more detail, consult the corresponding sections of the field report, QA report, or lab reports.

Purgeable Halocarbons:

Chloroform detected in both replicates of groundwater from A-2B from three analyses give consistent results: 11/54[a], 8.5/73[b], 13/140[c] ug/L. According to Chuck Stultz (DHS/DTSC-LA), there may have been some bailer malfunction during collection of the first sample, leading to loss of volatiles. The second sample may, therefore, be more representative of groundwater at A-2B.

Dichloromethane (Methylene Chloride) was detected in C-1B at 29[a]/24[b]/20[a] ug/L, in A-2M at 23[a]/25[c] ug/L, and in B-2M at 6.3[a]/3[c] ug/L and in a few other wells at close to the detection limit. Dichloromethane is a common lab solvent and often occurs as a random contaminant in samples. Because dichloromethane was detected in both VOA vial samples from the above wells, the results may be representative of the groundwater at these locations.

Tetrahydrofuran (THF) was detected in the sample from C-1B at 450[b] ug/L and in C-6B at 800[b] ug/L. THF was detected but not quantitated by method 624 at SRL. The THF results are consistent with earlier results from EPA and could be due to PVC glue solvent used in well construction.

1,1-Dichloroethane (1,1-DCA) was detected in C-5 at 5.2[c] ug/L, but as a rerun of this sample yielded less than the detection limit (<0.5 ug/L), the evidence is not conclusive for this compound.

Pesticides/PCBs:

No pesticides or PCBs were detected in any of the samples.

Total Organic Halogen (TOX):

The detection limits for TOX were so high that, while no TOX was detected reliably above the detection limit, low or moderate level contamination would not have been detected were it present.

Base/Neutral/Acid:

Phthalates were detected up to 50 ug/L in many of the wells. These are common contaminants, from contact with plastics. They may be present in the groundwater at these levels. No other B/N or A extractables were detected. Other, non- target compounds were tentatively identified, including 3- bromopentane, 4-chlorocyclohexanol, 3-bromocyclohexane, and 2,5- diethyltetrahydrofuran. If feasible, standards of these compounds should be obtained and used for comparison in future groundwater monitoring in order to confirm or refute their presence.

Metals:

Both dissolved Iron and Manganese are elevated in B-5, suggesting incomplete filtering of mineral material; Iron and Manganese are common constituents of soil minerals. As elevated concentrations of these elements occurs independently in other samples, their source is uncertain.

Dissolved copper appears to be elevated in B-3B. Dissolved selenium appears to be elevated in A-2M. Dissolved chromium was reported at 54 ug/L in A-2B. However, since it was neither detected in the duplicate sample nor in the total analysis above 4 ug/L, the value is not reliable.

General Inorganic:

C-1B showed highest pH, carbonate, and hydroxide values while showing low sulfate and

bicarbonate. These results are consistent with the low metals results for the same sample. These results may result from a poor seal between the well screening and the grout, or it could be due to contamination. B-3B showed the highest suspended solids, and nitrate values. The nitrate values exceed drinking water standard, but may be due to either formation or contaminants.

Conductivity is high in B-3M, B-5, and C-5. The latter two are collection (gallery) wells and are expected to catch contamination from the ponds. B-3M may reflect leachate (the only mantel well below B-5).

6.12 REFERENCES.

1) Laboratory Documentation Required for Data Evaluation, Quality Assurance Management Section, USEPA Region IX, R9QA/004.2, August 2001.

2) Principles of Environmental Analysis. American Chemical Society. Anal. Chem. 1983, 55, pp 2210-2218.

   An excellent discussion of environmental measurements including planning, sampling, analysis and reporting. Brief (9 pages) and readable; many valuable references are cited.

3) John Keenan Taylor, Quality Assurance of Chemical Measurements. Lewis Publishers, 1990, pp 7-10.

4) Helsel, Dennis R., Less than Obvious; Statistical Treatment of Data Below the Detection Limit, Environ. Sci. Technol., Vol 24, No. 12., pp 1766-1774, 1990.

7.0 GLOSSARY

AA: Atomic Absorption (Spectrometry). An instrument used to measure concentrations of metals in water, biological and soil samples (as extracts and digests). It is used in the less sensitive flame mode or the more sensitive furnace mode. It is adjusted to be selective for one single element at a time, as opposed to ICP.

Acceptable Criteria: specified limits placed on characteristics of an item, process, or service defined in requirement documents. (ASQC)

Accreditation: the process by which an agency or organization evaluates and recognizes a program of study or an institution as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory. In the context of the National Environmental Laboratory Accreditation Program (NELAP), this process is a voluntary one. (NELAC)

Accrediting Authority: the agency having responsibility and accountability for environmental laboratory accreditation and who grants accreditation. For the purposes of NELAC, this is EPA, other federal agencies, or the state. (NELAC)

Accuracy: the degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator. (Glossary of Quality Assurance Terms, QAMS, 8/31/92).

Analytical Reagent (AR) Grade: designation for the high purity of certain chemical reagents and solvents given the American Chemical Society. (Quality Systems)

Annular Space: The open space formed between the bore hole and the well casing. (i.e. outside the well casing)

Aquiclude: A geologic formation which may contain ground water but is incapable of transmitting significant quantities under normal hydraulic gradients.

Aquitard: A geologic formation of low permeability which can store or transmit ground water in significant quantities but typically at a very slow rate.

Assessor Body: the organization that actually executes the accreditation process, i.e., receives and reviews accreditation applications, reviews QA documents, reviews proficiency testing results, surveys the site, etc., whether EPA, the state, or contracted private party. (NELAP)

Audit: A systematic check to determine the quality of operation of some function or activity. Audits may be of two basic types: (1) performance audits in which quantitative or qualitative data are independently obtained for comparison with routinely obtained data in a measurement system, or (2) systems audits of a qualitative nature that consist of an on site review of a laboratory's quality assurance system and physical facilities for sampling, calibration, and measurement.

Background Sample: A sample that is taken from the general area where the sampling is being performed but remote from the actual sampling site. This sample should possess the same matrix characteristics as the actual samples, but be free of analytes. (Sometimes contain significant amounts of analyte, e.g., Pb in soil).

Bailer: A hollow, cylindrical device used to collect water samples. A ball valve allows water to enter from the bottom as the bailer is lowered, then prevents its release as the bailer is raised, thereby collecting the sample at desired depth.

Batch: environmental samples which are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents, with a maximum time between the start of processing of the first and last sample in the batch to be 24 hours. The size of a batch can range from one environmental sample to 20 environmental samples. All environmental samples in the batch must be of the same matrix as defined by NELAC. The resulting extracts, digestates or concentrates may be combined into an analytical batch. An analytical batch can include prepared samples originating from various environmental matrices and can exceed 20 samples. (Quality Systems)

Bentonite: A sedimentary rock largely comprised of clay minerals that have a great ability to absorb water and swell in volume.

Bias: The difference between the mean measurement and the reference or true value. Also see accuracy.

Bladder Pump: A device used to sample ground water. The pump uses a bag made of fluorocarbon material in order to prevent contamination of the sample and loss of volatile components.

Blank: a sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usually analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results. (ASQC, Definitions of Environmental Quality Assurance Terms, 1996)

Blind Sample: a subsample for analysis with a composition known to the submitter. The analyst/laboratory may know the identity of the sample but not its composition. It is used to test the analyst's or laboratory's proficiency in the execution of the measurement process.

Borehole Geophysics (Geophysical Borehole Logging): A general term that encompasses all techniques in which a sensing device is lowered into a borehole for the purpose of characterizing the associated geologic formations and their fluids. The results can be interpreted to determine lithology, geometry resistivity, bulk density, porosity, permeability, and moisture content and to define the source, movement, and physical/chemical characteristics of ground water.

CERCLA: Comprehensive Environmental Response, Compensation and Liability Act, PL 96-510, December 1980.

Calibrate: to determine, by measurement or comparison with a standard, the correct value of each scale reading on a meter or other devise, or the correct value for each setting of a control knob. The levels of the applied calibration standard should bracket the range of planned or expected sample measurements.

Calibration: the set of operations which establish, under specified conditions, the relationship between values indicated by a measuring instrument or measuring system, or values represented by a material measure, and the corresponding known values of a measurand. (VIM - 6.13)

Calibration Curve: the graphical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response.

Calibration Method: defined technical procedure for performing a calibration.

Calibration Standard: a solution prepared from the primary dilution standard solution or stock standard solutions and the internal standards and surrogate analytes. The Calibration solutions are used to calibrate the instrument response with respect to analyte concentration. (Glossary of Quality Assurance Terms, QAMS, 8/31/92).

Certified Reference Material (CRM): a reference material one or more of whose property values are certified by a technically valid procedure, accompanied by or traceable to a certificate or other documentation which is issued by a certifying body. (ISO Guide 30 - 2.2)

CFR: Code of Federal Regulation

Chain of Custody: an unbroken trail of accountability that insures the physical security of samples, data and records.

Chromatogram: A graph representing the signal output of an instrument (GC or HPLC) which can identify organic chemicals by peak retention time (RT) and quantitate by peak size.

CLP: EPA Contract Laboratory Program

Coefficient of Variation: A measure of relative dispersion. It is equal to the standard deviation divided by the mean and multiplied by 100 to give a percentage value. Also called relative standard deviation (RSD).

Co-located Sample (sometimes written collocated or collocated): Independent samples collected in such a manner that they are equally representative of the variable(s) of interest at a given point in space and time. Examples of collocated samples include: samples from two parallel samplers at the same location or two water samples collected at essentially the same time and from the same point in the lake. Results of collocated samples indicate the reproducibility (precision) of the sampling and analytical technique.

Comparability: A measure of the confidence with which one data set can be compared to another.

Completeness: A measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under correct and normal circumstances.

Compromised Samples: those samples which were improperly sampled, or with insufficient documentation (chain of custody and other sample records and/or labels), improper preservation and/or containers were used, or the holding time has been exceeded. Under normal conditions compromised samples are not analyzed. If emergency situations require analysis, the results must be appropriately qualified.

Concentration: The amount of chemical (analyte) present per amount of sample. For trace analyses, usually expressed as mg/L or ug/L for aqueous samples and mg/kg or ug/kg.

Cone of Depression: The cone-shaped lowering of the water table caused by pumping. Also referred to as drawdown.

Confined Aquifer: An aquifer under greater than atmospheric pressure bounded above and below by impermeable layer or layers of distinctly lower permeability (aquitard) than the aquifer itself.

Confirmation: verification of the presence of a component through the use of an analytical technique based on a different scientific principle from the original method. These may include:

Second column confirmation
Alternate wavelength
Derivatization
Mass spectral interpretation
Alternative detectors or
Additional cleanup procedures.

Corrective Action: action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation in order to prevent recurrence. (ISO 8402)

Coulson-GC: GC with a detector selective for halogenated organics, often used with the purge/trap technique for VOAs.

Data Reduction: the process of transforming raw data by arithmetic or statistical calculations, standard curves, concentration factors, etc., and collation into a more useful form.

Data Quality Objectives (DQOs): A statement of the precise data, the manner in which such data may be combined, and the acceptable uncertainty in those data in order to resolve an environmental problem or condition. This may also include the criteria or specifications needed to design a study that resolves the question or decision addressed by the DQO.

Data Audit: a qualitative and quantitative evaluation of the documentation and procedures associated with environmental measurements to verify that the resulting data are of acceptable quality (i.e., that they meet specified acceptance criteria.

Data Quality: The totality of features and characteristics of data that bears on its ability to satisfy a given purpose. The characteristics of major importance are accuracy, precision, existence of contamination, limit of detection completeness, representativeness, and comparability.

Data Validation: A systematic effort to review data to identify any outliers or errors and thereby cause deletion or flagging of suspect values to assure the validity of the data to the user. This auditing process may be done by manual and/or automated methods.

Depth to Bottom: Distance from fixed point, usually the top of the well casing, to the bottom of the well screen intake.

Depth to Water: Distance from a fixed point, usually the top of the well casing, to the top of the water surface.

Digestion: The process of extracting metals from a solid sample by heating with nitric acid.

Dioxin: Usually refers to 2,3,7,8-tetrachloro-p-dioxin (TCDD).

Dispersivity: Ability of a contaminant to disperse within the ground water due to molecular diffusion and mechanical mixing.

Document Control: the act of ensuring that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly and controlled to ensure use of the correct version at the location where the prescribed activity is performed. (ASQC, Definitions of Environmental Quality Assurance Terms, 1996)

Double Blind Sample: a sample submitted to evaluate performance with concentration and identity unknown to the analyst.

Downgradient: Direction of decreasing hydrostatic pressure.

Drilling Mud: Fluids which are used during the drilling of a borehole or well to wash soil cuttings away from the drill bit and adjust the specific gravity of the liquid in the borehole so that the sides of the hole do not cave in prior to installation of a casing.

Duplicate Analyses: the analyses or measurements of the variable of interest performed identically on two subsamples of the same sample. The results from duplicate analyses are used to evaluate analytical or measurement precision but not the precision of sampling, preservation or storage internal to the laboratory.

EC/GC: Electron-Capture Gas Chromatography. GC with a detector selective for halogenated organic chemicals (usually chlorinated hydrocarbon pesticides).

Elevation: Height relative to mean sea level.

Environmentally-Related Measurements: A term used to describe essentially all field and laboratory investigations that generate data involving the measurement of chemical, physical, or biological parameters in the environment; determining the presence or absence of priority pollutants in waste streams; health and ecological effect studies; clinical and epidemiological investigations; engineering and process evaluations; studies involving laboratory simulation of environmental events; and studies or measurements on pollutant transport, including diffusion models.

EP: EPA Extraction Procedure Toxicity Method: 40 CFR App. II, Apr. 8, 1983.

Equipment Blank: A sample that is made by collecting the final solvent rinsate used to rinse the sampling equipment.

Field Duplicate: Refers either to two separate samples collected from the same location in the field or to a single sample split into two portions in the field, preserved, transported, stored, prepared and analyzed identically, Results indicate the reproducibility (precision) of all the processes described above and analytical techniques.

Field Matrix Spike: A sample created by spiking target analytes into a portion of a sample in the field at the point of sample acquisition. This sample provides information on the target analyte stability and loss due to volatility after collection and during transport, storage, sample preparation and analysis.

GC: Gas Chromatograph - An instrument used to qualitatively and quantitatively identify volatile and semivolatile organic chemicals in a sample extract.

GC/MS: Gas Chromatography/Mass Spectrometer. GC with a mass spectrometric detector that provides almost absolute identification by taking a "fingerprint" (mass spectrum) of each organic chemical present in the sample.

Hall-GC: GC with a detector selective for halogenated organics, often used with the purge/trap technique for VOAs.

Holding Times (Maximum Allowable Holding Times): the maximum times that samples may be held prior to analysis and still be considered valid. (40 CFR Part 136).

HPLC: High Performance Liquid Chromatography. A chromatograph which is used to qualitatively and quantitatively identify organic chemicals, particularly those which are not amenable to GC techniques because of thermal instability, polarity or nonvolatility.

Hydraulic Conductivity: a coefficient of proportionality which describes the rate at which a fluid can move through a permeable medium such as an aquifer. It is a function of the media and of the fluid flowing through it.

ICP (ICPAES): Inductively Coupled Plasma Atomic Emission Spectroscopy. An instrument used to measure concentrations of metals in water samples, extracts and digests. The instruments are either simultaneous or sequential and are capable of measuring the presence and amount of a variety of metals at one time.

Initial Demonstration of Analytical Capability: procedure to establish the ability to generate acceptable accuracy and precision which is included in many of the EPA's

analytical methods. In general the procedure includes the addition of a specified concentration of each analyte (using a QC check sample) in each of four separate aliquots of laboratory pure water. These are carried through the entire analytical procedure and the percentage recovery and the standard deviation are determined and compared to specified limits. (40 CFR Part 136).

Instrument Blank: a clean sample (e.g., distilled water) processed through the instrumental steps of the measurement process; used to determine instrument contamination. (Glossary of Quality Assurance Terms, QAMS, 8/31/92).

Interface Probe: Instrument capable of detecting an immiscible organic layer floating on the surface of the water.

Internal Standard: a known amount of standard added to a test portion of a sample and carried through the entire measurement process as a reference for evaluating and controlling the precision and bias of the applied analytical method.

Intrinsic Permeability: relates to the relative ability of a porous medium to transmit liquid under a hydraulic gradient, and is independent of the liquid itself.

It is also expressed as the degree of agreement of a measurement (or an average of measurements of the same thing), X, with an accepted reference or true value, T, usually expressed as the difference between two values, X- T, or the difference as a percentage of the reference or true value, 100(X-T)/T, and sometimes expressed as a ratio, X/T. Accuracy is a measure of the bias in a system.

Laboratory Matrix Spike: A sample created by spiking target compounds into a portion of a sample when it is received in the laboratory. It provides information on the analytical accuracy of sample preparation and analysis and is the most common type of spiked sample. A lab matrix spike does not necessarily reflect the behavior of the field-collected target analyte, especially if the target analyte is not stable during shipping.

Laboratory: Body that calibrates and/or tests.

NOTES:

1. In cases where a laboratory forms part of an organization that carries out other activities besides calibration and testing, the term "laboratory" refers only to those parts of that organization that are involved in the calibration and testing process.

2. As used herein, the term "laboratory" refers to a body that carries out calibration or testing
   - at or from a permanent location,

- at or from a temporary facility, or
- in or from a mobile facility. (ISO 25)

Laboratory Control Sample (quality control sample): an uncontaminated sample matrix spiked with known amounts of analytes from a source independent of the calibration standards. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system. (Glossary of Quality Assurance Terms, QAMS, 8/31/92).

Laboratory Duplicate: Aliquots of a sample taken from the same container under laboratory conditions and processed and analyzed independently.

Leachate: A liquid including any suspended components in the liquid that has percolated through or drained from hazardous waste.

Legal Chain of Custody (COC): an unbroken trail of accountability that ensures the physical security of samples, data and records. (Glossary of Quality Assurance Terms, QAMS, 8/31/92).

Limit of detection: Limit at which an analyte can be reported to be present at a specified confidence level.

Limit of quantitation: Limit at which the concentration of an analyte is reported at a specified confidence level.

Manager (however named): the individual designated as being responsible for the overall operation, all personnel, and the physical plant of the environmental laboratory. A supervisor may report to the manager. In some cases, the supervisor and the manager may be the same individual.

Matrix: The component or substrate which contains the analyte of interest. For purposes of batch determination, the following matrix types shall be used:

Drinking water:	Any aqueous sample that has been designated as a potable or potential potable water source.
Aqueous:	Any aqueous sample excluded from the definition of a water matrix or Saline/Estuarine source. Includes surface water, groundwater and effluents.
Saline/Estuarine:	Any aqueous sample from an ocean or estuary, or other salt water source such as the Great Salt Lake.

Non-aqueous liquid:	Any organic liquid with <15% settleable solids.
Biological Tissue:	Any sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples shall be grouped according to origin.
Solids:	Includes soils, sediments, sludges and other matrices with >15% settleable solids.
Chemical Waste:	A product or by-product of an industrial process that results in a matrix not previously defined.
Air Samples:	Media used to retain the analyte of interest from an air sample such as sorbent tubes or summa canisters. Each medium shall be considered as a distinct matrix. (Quality Systems)

Matrix Spike (spiked sample, fortified sample): prepared by adding a known mass of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency. (Glossary of Quality Assurance Terms, QAMS, 8/31/92).

Matrix Spike Duplicate (spiked sample/fortified sample duplicate): a second replicate matrix spike is prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte. (Glossary of Quality Assurance Terms, QAMS, 8/31/92).

May: permitted, but not required (TRADE)

Median: The middle point in a set of measurements ranked by numerical value. Half of the numbers lie above and half below the median. If there is an even number of measurements, the medium is the mean of the two central measurements.

Method Blank: a clean sample processed simultaneously with and under the same conditions as samples containing an analyte of interest through all steps of the analytical procedures. (Glossary of Quality Assurance Terms, QAMS, 8/31/92).

Method Detection Limit (Analytical Detection Limit): the minimum concentration of a substance (an analyte) that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte. (40 CFR Part 136 Appendix B).

Method Standard: The solvent is spiked with analytes of interest from an independent source to monitor the analytical method. These standards are used to document interference free recoveries.

Mounding: A phenomenon usually created by the recharge of ground water from a manmade structure into a permeable geologic material. Associated ground-water flow will be away from the manmade structure in all directions.

Must: denotes a requirement that must be met. (Random House College Dictionary)

Negative Control: measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results.

NELAC: National Environmental Laboratory Accreditation Conference. A voluntary organization of state and federal environmental officials and interest groups purposed primarily to establish mutually acceptable standards for accrediting environmental laboratories. A subset of NELAP. (NELAC)

NELAP: the overall National Environmental Laboratory Accreditation Program of which NELAC is a part. (NELAC)

Octanol-Water Partition Coefficient: A coefficient representing the ratio of solubility of a compound in octanol to its solubility in water. As the octanol-water partition coefficient increases, water solubility decreases.

Organics: Most chemicals that contain the element carbon are organic chemicals or "organics". Organic chemicals can be synthetic or derived from natural sources. Pesticides and priority pollutants are examples of hazardous organics.

OVA: Organic Vapor Analyzer - A handheld air monitoring instrument which detects a wide range of organic vapors through flame ionization.

PAHs (PNAs): Polynuclear Aromatic Hydrocarbons. A class of hydrocarbons based on combinations of benzene rings. Also called PNAs (polynuclear aromatics).

Parameter: A constant or coefficient that describes some characteristic of a population (e.g., standard deviation, mean, regression coefficients). At times this term is used interchangeably with analyte.

PCBs: Polychlorinated Biphenyls, a class of chlorinated organic mixtures previously used as insulators in transformers. The four most common mixtures are called Aroclors 1242, 1248, 1254, and 1260. These designations represent the number of carbon atoms (12) and percent weight chlorine 42) for Arochlor 1242.

Performance Audit: the routine comparison of independently obtained quantitative measurement system data with routinely obtained data in order to evaluate the proficiency of an analyst or laboratory.

Performance Based Measurement System (PBMS): a set of processes wherein the data quality needs, mandates or limitations of a program or project are specified and serve as criteria for selecting appropriate methods to meet those needs in a cost-effective manner.

Phreatic Zone: See Saturated Zone

Piezometers: Generally a small diameter, non-pumping well used to measure the elevation of the water table or potentiometric surface.

Positive Control: measures taken to ensure that a test and/or its components are working properly and producing correct or expected results from positive test subjects.

Potentiometric Surface (Piezometric Surface): The surface that represents the level to which water from a given aquifer will rise by hydrostatic pressure. When the water-bearing zone is the uppermost unconfined aquifer, the potentiometric surface is identical to the water table.

Precision: the degree to which a set of observations or measurements of the same property, usually obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms. (Glossary of Quality Assurance Terms, QAMS, 8/31/92).

Preservation: refrigeration and or reagents added at the time of sample collection to maintain the chemical and or biological integrity of the sample.

Proficiency Testing: Determination of the laboratory calibration or testing performance by means of interlaboratory comparisons. (ISO/IEC Guide 2 - 12.6, amended)

Proficiency Test Sample (PE): a sample, the composition of which is unknown to the analyst and is provided to test whether the analyst/laboratory can produce analytical results within specified performance limits. (Glossary of Quality Assurance Terms, QAMS, 8/31/92).

Protocol: a detailed written procedure for field and/or laboratory operation (e.g., sampling, analysis) which must be strictly followed.

Pump Test: A test made by pumping a well for a period of time and observing the change in hydraulic head in adjacent wells. A pump test may be used to determine degree of

hydraulic interconnection between different water-bearing units as well as the recharge rate of a well.

Pure Reagent Water: shall be ASTM Type I or Type II water in which no target analytes or interferences are detected as required by the analytical method.

Purgeables: Those volatile organic chemicals which are best analyzed by purging from a sample matrix.

QA/QC: Quality Assurance/Quality Control

Quality Assurance: an integrated system of activities involving planning, quality control, quality assessment, reporting and quality improvement to ensure that a product or service meets defined standards of quality with a stated level of confidence. (Glossary of Quality Assurance Terms, QAMS, 8/31/92).

Quality Control: the overall system of technical activities whose purpose is to measure and control the quality of a product or service so that it meets the needs of users. (Glossary of Quality Assurance Terms, QAMS, 8/31/92).

Quality Control Sample: an uncontaminated sample matrix spiked with known amounts of analytes from a source independent from the calibration standards. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system. (Glossary of Quality Assurance Terms, QAMS, 8/31/92).

Quality Manual: A document stating the quality policy, quality system and quality practices of an organization. This may be also called a Quality Assurance Plan or a Quality Plan.

> NOTE - The quality manual may call up other documentation relating to the laboratory's quality arrangements.

Quality System: a structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA and QC. (ANSI/ASQC E-41994)

Quality Assurance Project Plan: An orderly assembly of detailed and specific procedures by which an agency or laboratory delineates how it produces quality data for a specific project or measurement method. Sampling plans also should address quality assurance concerns for the sampling activities.

Quality Management Plan (QMP): It defines an organization's QA - related policies, criteria for and areas of application, and definition of roles, responsibilities, and authorities.

Range: the difference between the minimum and the maximum of a set of values.

RCRA: The federal Resource Conservation and Recovery Act, PL 94-590, October 1976.

Raw Data: any original factual information from a measurement activity or study recorded in a laboratory notebook, worksheets, records, memoranda, notes ,or exact copies thereof and that are necessary for the reconstruction and evaluation of the report of the activity or study. Raw data may include photography, microfilm or microfiche copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments. If exact copies of raw data have been prepared (e.g., tapes which have been transcribed verbatim, data and verified accurate by signature), the exact copy or exact transcript may be submitted.

Reagent Blank (method reagent blank): a sample consisting of reagent(s), without the target analyte or sample matrix, introduced into the analytical procedure at the appropriate point and carried through all subsequent steps to determine the contribution of the reagents and of the involved analytical steps. (Glossary of Quality Assurance Terms, QAMS, 8/31/92).

Reference Material: a material or substance one or more properties of which are sufficiently well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials. (ISO Guide 30 - 2.1)

Reference Standard: a standard, generally of the highest metrological quality available at a given location, from which measurements made at that location are derived. (VIM - 6.08)

Reference Toxicant: see D.2.1.a

Replicate Analyses: the measurements of the variable of interest performed identically on two or more subsamples of the same sample within a short time interval.

Representativeness: the degree to which data accurately and precisely represents a characteristic of a population, the variation of a parameter at a sampling point, or an environmental condition.

Requirement: a translation of the needs into a set of individual quantified or descriptive specifications for the characteristics of an entity in order to enable its realization and examination.

Sample Duplicate: two samples taken from and representative of the same population and carried through all steps of the sampling and analytical procedures in an identical manner. Duplicate samples are used to assess variance of the total method including sampling and analysis. (Glossary of Quality Assurance Terms, QAMS, 8/31/92).
Sampling Spike: A compound that is not present in the environment added to the filter or sorbent bed to study the sampling efficiency.

Saturated Zone (Phreatic Zone): A subsurface zone in which the pore space is completely filled with water.

Seep: A spot where groundwater trickles out of the ground.
Significant Figures - Digits in a number that have some practical meaning. For a number with significant figures, all digits are certain except for the last digit, which may be in doubt.

Selectivity: (Analytical chemistry) the capability of a method or instrument to respond to a target substance or constituent in the presence of nontarget substances.

Sensitivity: the capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest.

Shall: denotes a requirement that is mandatory whenever the criterion for conformance with the specification requires that there be no deviation. This does not prohibit the use of alternative approaches or methods for implementing the specification so long as the requirement is fulfilled. (*Style Manual for Preparation of Proposed American National Standards*, American National Standards Institute, eighth edition, March 1991).

Should: denotes a guideline or recommendation whenever noncompliance with the specification is permissible. (*Style Manual for Preparation of Proposed American National Standards*, American National Standards Institute, eighth edition, March 1991).

Slug Test: An aquifer test made by either pouring a small charge of water into a well or by withdrawing a slug of water from the well and monitoring the length of time the well requires to return to static water level conditions. This test is often employed to determine hydraulic conductivity.

Solvent Blank: A sample made by taking a portion of the solvent used to extract a sample.

Specific Conductance: Specific Conductance (or Electrical Conductivity) is a gross analytical test for the presence of inorganic chemicals.

Spike: a known mass of target analyte added to a blank sample or subsample; used to determine recovery efficiency or for other quality control purposes.

Split Sample: a homogenized sample divided into two portions, which are usually sent to different organizations or laboratories and subjected to the same environmental conditions and steps in the measurement process.

Standard Operating Procedures (SOPs): a written document which details the method of an operation, analysis or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks. (Glossary of Quality Assurance Terms, QAMS, 8/31/92).

Standard Reference Material (SRM): a certified reference material produced by the U.S. National Institute of Standards and Technology and characterized for absolute content, independent of analytical method.

Standard Deviation (s): a measure of the dispersion about the mean of the elements in a population.

Standing Water: groundwater standing in a well which is not being pumped.

STLC: Soluble Threshold Limit Concentration. As defined by the WET, the maximum leachable concentrations of chemicals allowed in a non-hazardous waste. See TTLC.

Supervisor (however named): the individual(s) designated as being responsible for a particular area or category of scientific analysis. This responsibility includes direct day-to-day supervision of technical employees, supply and instrument adequacy and upkeep, quality assurance/quality control duties and ascertaining that technical employees have the required balance of education, training and experience to perform the required analyses.

Surrogate: a substance with properties that mimic the analyte of interest. It is unlikely to be found in environment samples and is added to them for quality control purposes. (Glossary of Quality Assurance Terms, QAMS, 8/31/92).

Systems Audit (also Technical Systems Audit): a thorough, systematic on-site, qualitative review of the facilities, equipment, personnel, training, procedures, record keeping, data validation, data management, and reporting aspects of a total measurement system.

Technical Analyst: the designated individual who performs the "hands-on" analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent Quality Controls to meet the required level of quality.

Test Method: defined technical procedure for performing a test.

Test: a technical operation that consists of the determination of one or more characteristics or performance of a given product, material, equipment, organism, physical phenomenon, process or service according to a specified procedure.

NOTE - The result of a test is normally recorded in a document sometimes called a test report or a test certificate. (ISO/IEC Guide 2 - 12.1, amended)

Testing Laboratory: laboratory that performs tests. (ISO/IEC Guide 2 - 12.4)

The media in which analytes are tested at ECL includes air, water, soil and solids. Water and soil matrices are the most commonly encountered matrices. Therefore in defining the matrices for the purpose of performing the required matrix spike and matrix spike duplicates these two matrix types, water and soil, are considered independent and all samples analyzed should be broadly classified into one of these two matrix types.

TOC: Total Organic Carbon (SW-846, Method 9060).

TOX: Total Organic Halogens (SW-846, Method 9020).

Total Quality Management (TQM): The process of applying quality management to all activities of the organization, including technical and administrative operations.

Traceability: the property of a result of a measurement whereby it can be related to appropriate standards, generally international or national standards, through an unbroken chain of comparisons. (VIM - 6.12)

Travel Blank (Trip Blank): A sample, usually purified (organic free) water, prepared in the laboratory, which is taken to the sampling site and then returned with the collected samples. Later analysis will eliminate any false positive results in the real samples arising from contamination during shipment. Also called Trip Blank. See also Trip Spike.

Trip Spike: A sample, usually water, to which a known amount of the chemical of interest is added in the lab before a sampling trip. Later analysis will eliminate any false negative results in the real samples arising from degradation during shipment. See also Travel Blank.

TTLC: Total Threshold Limit Concentration. As defined by the WET, the maximum total concentrations of chemicals allowed in a non-hazardous waste. See STLC.

Turbidity: refers to the decrease in transparency of the water due to suspended particles.

Unsaturated Zone: (Vadose Zone) - a subsurface zone above the water table in which the

soil pores of a porous medium are only partially filled with water.

Upgradient: direction of increasing hydrostatic pressure.

Vadose Zone: See Unsaturated Zone.

Validation: the process of substantiating specified performance criteria.

Verification: confirmation by examination and provision of evidence that specified requirements have been met.

NOTE - In connection with the management of measuring equipment, verification provides a means for checking that the deviations between values indicated by a measuring instrument and corresponding known values of a measured quantity are consistently smaller than the maximum allowable error defined in a standard, regulation or specification peculiar to the management of the measuring equipment.

The result of verification leads to a decision either to restore in service, to perform adjustments, or to repair, or to downgrade, or to declare obsolete. In all cases it is required that a written trace of the verification performed shall be kept on the measuring instrument's individual record.

VOA (VOC): Volatile Organic Analyses (Volatile Organic Carbon). A test for volatile organic chemicals by the purge and trap technique using GC with a halide-specific detector (e.g., Coulson or Hall for chlorinated, brominated or fluorinated volatile organics) or with GC/MS (for any volatile organic). Mainly used for trihalomethanes or industrial solvents in groundwater samples.

Volatile Constituents: solid or liquid compounds which are relatively unstable at standard temperature and pressure and undergo spontaneous phase change to a gaseous state. Vapor pressure is a measure of a compound's volatility in its pure phase. Henry's constant is measure of its volatility from aqueous solution.

Water Table: the water level surface below the ground at which the vadose zone ends and the phreatic zone begins. It is the level to which a well screened in the unconfined aquifer would fill with water.

Well Casing: rigid cylindrical material inserted into the well borehole.

Well Screen: the perforated section of the well casing.

Well Volume (V): volume of standing water in a well calculated as $V=(PI)*V^2*$(height of the water column)

APPENDICES

1.0 Sample Analysis Authorizations and Analysis Requests

One of ECL's primary roles is to analyze samples collected by DTSC field staff. If ECL doesn't have the capability to do a specific analysis, or to analyze samples within the time requested, ECL will arrange for a Contract Laboratory to do the analysis. The Contract Laboratory will be accredited by the National Environmental Laboratory Accreditation Program (NELAP). ECL will coordinate analysis with the Contract Laboratory and perform a quality assurance review of the completed laboratory report, and if needed the data package.

The ECL Sample Management Officer (SMO) will be the main point of contact on matters regarding sample analysis between DTSC programs and ECL, and between DTSC and the Contract Laboratories. The SMO, calling on resources throughout ECL, can help the field staff ("Requestor") develop a Sampling and Analysis Plan. The SMO can assist with the selection of analytical methods, sample containers, preservatives, and the amount of sample required for the analyses requested. The SMO will survey available laboratory resources, and direct samples to one or more laboratories that have the ability to complete the analysis within the holding times stipulated in the method, and within the turn around time required by the Requestor.

There are four turnaround time (TAT) levels. The quickest is TAT 1, 15 days or less between sample arrival and the laboratory report. TAT 1 requires approval of the Unit Chief. In an emergency, sample analysis can be expedited. The program Branch Chief should contact the ECL Chief directly.

ECL can also accommodate non-routine analysis requests and conduct special projects.
Contact the Research Scientist Supervisor or the ECL Chief directly.

The procedures for obtaining approval for sample analysis and for submitting samples are detailed in this Appendix. The next two sections describe the information and sample flow, and specifically the two forms (ARF and SAR) that serve to initiate and document the sampling event and subsequent analysis. The ARF and SAR, and instructions for completing them, are on the DTSC network server so they may be readily accessed, completed and transmitted on-line.

2.0 Authorization Request Form (ARF) for Sample Analysis

2.1 At least one week before the anticipated sample collection date, the Requestor will complete Parts A and B of the electronic Authorization Request Form (e-ARF). The e-ARF Excel file and instructions for completing the form are on the DTSC shared drive forms directory T: \FORMS\ECL\ARF. The Requestor should contact the ECL Sample Management Officer (SMO) with any questions regarding the form, including the selection of analytical methods. Each sampling event requires its own ARF.

2.2. The ARF file is e-mailed to the ECL SMO GroupWise mailbox at: SMOff@dtsc.ca.gov.

2.3 The SMO will review the ARF for completeness and, if necessary, contact the Requestor for clarification. Substantial changes will be noted as a revision in Part C of the ARF.

2.4. The SMO will e-mail the ARF to the appropriate ECL supervisors in Berkeley (for ARFs submitted by DTSC HQ, Region 1 or Region 2) or Los Angeles (for ARFs submitted by Region 3 or Region 4).

2.5. The ECL supervisors will determine if they will have the capacity (available staff and equipment) to do the requested analyses after the expected sample arrival date, and before the sample holding time prescribed for each method or the requested turn around time has lapsed.

2.6 The ECL supervisors will accept or reject all or part or the analyses requested. They will inform the SMO of their decision by e-mail within 24 hours.

2.7. If the analysis can be done in-house, the samples will be directed to the appropriate ECL (Berkeley or Los Angeles). If the capacity does not exist at ECL, the samples will be assigned to the closest DTSC contract laboratory. Samples and analyses may be divided between the two ECL laboratories, or an ECL and a contract laboratory. The SMO, in consultation with the Requestor, the ECL laboratory supervisors, and the contract laboratory project managers, will determine the optimal distribution of samples and analyses.

2.8. The SMO will complete Part C of the ARF, assigning an Authorization Number (AN), a laboratory to receive the samples, and the Expiration Date for the request. The AN comprises two numbers and two letters that denote the fiscal year and the assigned laboratory, respectively, and a four-digit sequential number that resets to 0001 on July 1. (For example, the first AN assigned after July 1, 2007 for samples to be analyzed at ECL-Berkeley will be 07EC0001. The code for ECL-Los Angeles will be 07SC0001.) The Requestor will enter the AN on the Sample Analysis Request (SAR) form completed at the time of sampling. The Requestor should refer to the AN when contacting the laboratories regarding the samples.

2.9 If samples are to be sent to two laboratories, an additional ARF will be created by copying the submitted e-ARF. The SMO will modify Part B of the two ARFs (the original and copy) so that only the samples and analyses to be done at the assigned laboratory in Part C will appear on the copy sent to that laboratory.

2.10 The SMO will transmit by e-mail the completed ARF(s) to the Requestor, the appropriate ECL supervisors, and the contract laboratory, as indicated.

2.11 The SMO will archive the ARF in electronic file format.

2.12 If samples and an e-SAR are not received within a week after the Expiration Date, the Requestor will receive a follow-up reminder by e-mail.

3.0 Sample Analysis Requests (SAR)

3.1 After an approved Authorization Request Form (ARF) has been received, samples may be delivered to the designated laboratory for analysis. Because of the time required to process ARFs, it is advisable to collect samples only after ARF approval is obtained and the laboratory is prepared to receive the samples. This way, unnecessary delays between sample collection and analysis that may compromise sample integrity and data quality can be avoided. In the event of an emergency or other extenuating circumstance, field staff can contact the ECL Sample Management Officer (SMO) and request expedited processing of the ARF. The number of samples, analyses requested, and sampling date should conform to the ARF. If the number of samples is 20 percent or more than

what was approved, if additional analyses are required, or if sampling will be delayed or cancelled, the Requestor should inform the SMO and submit a revised ARF.

3.2 During sample collection, field data should be recorded for entry on the electronic Sample Analysis Request (e-SAR) form. This information can be directly entered on a portable computer carried into the field, or recorded in a notebook or printed copy of the SAR and entered later into the e-SAR. The e-SAR (in EXCEL) and instructions for completing the form are on the DTSC shared drive forms directory T:\FORMS\ECL\SAR. Many of the fields have drop-down menus; others require the Requestor to enter the information.

3.3 All SARs should be e-mailed to the ECL Sample Log-in Officer (SLOff) at SLOff@dtsc.ca.gov so all samples, including those going to contract laboratories, can be tracked in the ECL Laboratory Information Management System (LIMS).

3.4 The SLOff will forward the e-SARs to ECL-Los Angeles for samples that will be delivered there.

3.5 The Requestor should print a copy of the completed SAR and fill in line "a" of the Chain of Custody (CoC) section of the SAR. CoC documentation must start at the time of sample collection. A copy of this signed SAR must accompany the samples to the laboratory.

3.6 The ECL SLOff will fill in the next line of the CoC on the SAR, acknowledging receipt of the samples, and return a copy to the Requestor.

3.7 If samples will be forwarded from ECL to a contract lab, a copy of the SAR with CoC will be sent with the samples.

3.8 If samples will be sent directly to a contract lab by the Requestor, a copy of the signed SAR with CoC should be sent with the samples. The Requestor should also e-mail the unsigned e-SAR to the SLOff. This can be done directly from the form if using a DTSC-network computer.

PARAMETER	LO STD CONC (ug/l)	ECL WATER[a] (ug/l)	ECL SOIL[b] (mg/kg)	LO STD CONC (DRINKING WATER ONLY) (ug/l)	DRINKING WATERc (ug/l)
Bromomethane	5.0	5.0	0.6	0.5	0.5
Vinyl chloride	5.0	5.0	0.6	0.5	0.5
Chloroethane	5.0	5.0	0.6	0.5	0.5
Methylene chloride	5.0	5.0	0.6	0.5	0.5
Trichlorofluoromethane	5.0	5.0	0.6	0.5	0.5
1,1-Dichloroethene	5.0	5.0	0.6	0.5	0.5
1,1-Dichloroethane	5.0	5.0	0.6	0.5	0.5
trans 1,2-Dichloroethene	5.0	5.0	0.6	0.5	0.5
Chloroform	5.0	5.0	0.6	0.5	0.5
1,2-Dichloroethane	5.0	5.0	0.6	0.5	0.5
1,1,1-Trichloroethane	5.0	5.0	0.6	0.5	0.5
Carbon tetrachloride	5.0	5.0	0.6	0.5	0.5
Bromodichloromethane	5.0	5.0	0.6	0.5	0.5
1,2-Dichloropropane	**5.0**	**5.0**	**0.6**	**0.5**	**0.5**
cis-1,3-Dichloropropene	5.0	5.0	0.6	0.5	0.5
Trichloroethylene	5.0	5.0	0.6	0.5	0.5
Dibromochloromethane	5.0	5.0	0.6	0.5	0.5
1,1,2-Trichloroethane	5.0	5.0	0.6	0.5	0.5
trans-1,3-Dichloropropene	5.0	5.0	0.6	0.5	0.5
1,1,2,2-Tetrachloroethane	5.0	5.0	0.6	0.5	0.5
Tetrachloroethylene	5.0	5.0	0.6	0.5	0.5
Chlorobenzene	5.0	5.0	0.6	0.5	0.5
Benzene	5.0	5.0	0.6	0.5	0.5
1,2-Dichlorobenzene	5.0	5.0	0.6	0.5	0.5
1,3-Dichlorobenzene	5.0	5.0	0.6	0.5	0.5
1,4-Dicholorbenzene	5.0	5.0	0.6	0.5	0.5
Ethylbenzene	5.0	5.0	0.6	0.5	0.5
Toluene	5.0	5.0	0.6	0.5	0.5
Xylenes	5.0	5.0	0.6	0.5	0.5
Bromochloromethane	5.0	5.0	0.6	0.5	0.5
Bromoform	5.0	5.0	0.6	0.5	0.5
4Chlorotoluene	5.0	5.0	0.6	0.5	0.5
1,2-Dibromo-3-chloropropane	5.0	5.0	0.6	0.5	0.5
1,2-dibromomethane	5.0	5.0	0.6	0.5	0.5
Dibromomethane	5.0	5.0	0.6	0.5	0.5
Dichlorodofloromethane	5.0	5.0	0.6	0.5	0.5
cis-1,2-Dichloroethene	5.0	5.0	0.6	0.5	0.5
1,3-Dichlor-2-propanol	5.0	5.0	0.6	0.5	0.5
Hexachlorobutadiene	5.0	5.0	0.6	0.5	0.5
Naphthalene	5.0	5.0	0.6	0.5	0.5
Styrene	5.0	5.0	0.6	0.5	0.5
1,2,4-Trichlorobenzene	5.0	5.0	0.6	0.5	0.5
1,2,3-Trichloropropane	5.0	5.0	0.6	0.5	0.5

Table 1. Routine Quantitation Limits for Analysis of Aromatic and Halogenated Volatile Organics (EPA 8021B).

a Direct Purge Dilution Factor **(DF)** = **1** (Water)
b Extraction Method: EPA 5030 4 g → 10 mL = **(1)** 100 uL of **(1)** → 5 mL ∴ Dilution Factor **(DF) = 125** (Soil)
c Direct Purge Dilution Factor **(DF)** = **1** (Drinking Water)

PARAMETER	LO STD CONC (ug/l)	ROUTINE WATER[a] (ug/l)	ROUTINE SOIL[b] (mg/kg)	LO WATER[c] (ug/l)	LO SOIL[d] (mg/kg)	HI WATER&SOILe	DRINKING WATERf (ug/l)
			ECL				
Delta-BHC	10	10	0.10	0.10	0.033	.....	0.020
A-BHC	10	10	0.10	0.10	0.033	.....	0.020
B-BHC	10	10	0.10	0.10	0.033	.....	0.020
Lindane	10	10	0.10	0.10	0.033	.....	0.020
PCNB	10	10	0.10	0.10	0.033	.....	0.020
Heptachlor	10	10	0.10	0.10	0.033	.....	0.020
Aldrin	10	10	0.10	0.10	0.033	.....	0.020
Heptachlor epoxide	10	10	0.10	0.10	0.033	.....	0.020
A-Chlordane	10	10	0.10	0.10	0.033	.....	0.020
2,4'-DDE	10	10	0.10	0.10	0.033	.....	0.020
Endosulfan I	10	10	0.10	0.10	0.033	.....	0.020
gamma-Chlordane	10	10	0.10	0.10	0.033	.....	0.020
4,4'-DDE	10	10	0.10	0.10	0.033	.....	0.020
Endosulfan sulfate	10	10	0.10	0.10	0.033	.....	0.020
Endrin aldehyde	10	10	0.10	0.10	0.033	.....	0.020
Dieldrin	10	10	0.10	0.10	0.033	.....	0.020
2,4'-DDD	10	10	0.10	0.10	0.033	.....	0.020
Endrin	10	10	0.10	0.10	0.033	.....	0.020
Endosulfan II	10	10	0.10	0.10	0.033	.....	0.020
4,4'-DDD	10	10	0.10	0.10	0.033	.....	0.020
2,4-'DDT	10	10	0.10	0.10	0.033	.....	0.020
4,4'-DDT	10	10	0.10	0.10	0.033	.....	0.020
4,4'-Methoxychlor	40	40	0.40	0.40	0.13	.....	0.080
Tedion	20	20	0.20	0.20	0.067	.....	0.040
Mirex	10	10	0.10	0.10	0.033	.....	0.020
Toxaphene	250	250	2.5	2.5	0.87	.....	0.50
PCB 1016	200	200	2.0	2.0	0.2	.....	0.20
PCB 1221	200	200	2.0	2.0	0.2	.....	0.20
PCB 1232	200	200	2.0	2.0	0.2	.....	0.20
PCB 1242	200	200	2.0	2.0	0.2	.....	0.20
PCB 1248	200	200	2.0	2.0	0.2	.....	0.20
PCB 1254	200	200	2.0	2.0	0.2	.....	0.20
PCB 1260	200	200	2.0	2.0	0.2	.....	0.20
PCB 1262	200	200	2.0	2.0	0.2	.....	0.20
Toxaphene	250	250	2.5	2.5	0.87	.....	0.50
PCB 1016	200	200	2.0	2.0	0.2	.....	0.20
PCB 1221	200	200	2.0	2.0	0.2	.....	0.20

Table 2a. Quantitation Limits for Analysis of Chlorinated Pesticides (EPA 8081/8082).

a 100 mL → 100 mL ∴ **DF = 1.0** (Routine Water)
b 10 g → 100 mL ∴ **DF = 10.0** (Routine Soil)
c 1000 mL → 10 mL ∴ **DF = 0.01** (Low Level Water)
d 30 g → 100 mL ∴ **DF = 3.33** (Low Level Soil)
e Dependent on Matrix
f 1000 mL → 2 mL ∴ **DF = 0.002** (Drinking Water)

ECL-SC

PARAMETER	LO STD CONC (ug/l)	ROUTINE WATER[a] (ug/l)	ROUTINE SOIL[b] (mg/kg)	LO WATER[c] (ug/l)	LO SOIL[d] (ug/kg)	HI WATER&SOIL[e]
Delta-BHC	5.0	0.25	0.050	0.050	5.0	.....
a-BHC	5.0	0.25	0.050	0.050	5.0	.....
b-BHC	5.0	0.25	0.050	0.050	5.0	.....
Lindane	5.0	0.25	0.050	0.050	5.0	.....
PCNB	.....	.....	.....	.....	.....	.....
Heptachlor	5.0	0.25	0.050	0.050	5.0	.....
Aldrin	5.0	0.25	0.050	0.050	5.0	.....
Heptachlor epoxide	5.0	0.25	0.050	0.050	5.0	.....
a-Chlordane	.....	.....	.....	.....	.....	.....
o,p'-DDE	.....	.....	.....	.....	.....	.....
Endosulfan I	5.0	0.25	0.050	0.050	5.0	.....
gamma-Chlordane	.....	.....	.....	.....	.....	.....
p,p'-DDE	5.0	0.25	0.050	0.050	5.0	.....
Endosulfan sulfate	.....	.....	.....	.....	.....	.....
Endrin aldehyde	.....	.....	.....	.....	.....	.....
Dieldrin	5.0	0.25	0.050	0.050	5.0	.....
o,p'-DDD	.....	.....	.....	.....	.....	.....
Endrin	5.0	0.25	0.050	0.050	5.0	.....
Endosulfan II	5.0	0.25	0.050	0.050	5.0	.....
p,p'-DDD	5.0	0.25	0.050	0.050	5.0	.....
o,p-'DDT	.....	.....	.....	.....	.....	.....
p,p'-DDT	5.0	0.25	0.050	0.050	5.0	.....
p,p'-Methoxychlor	5.0	0.25	0.050	0.050	5.0	.....
Tedion	.....	.....	.....	.....	.....	.....
Mirex	.....	.....	.....	.....	.....	.....
Toxaphene	250	12	2.5	2.5	250	.....
PCB 1016	500	25	0.50	5.0	500	.....
PCB 1221	500	25	0.50	5.0	500	.....
PCB 1232	500	25	0.50	5.0	500	.....
PCB 1242	500	25	0.50	5.0	500	.....
PCB 1248	500	25	0.50	5.0	500	.....
PCB 1254	500	25	0.50	5.0	500	.....
PCB 1260	500	25	0.50	5.0	500	.....
PCB 1262	500	25	0.50	5.0	500	.....

Table 2b. Quantitation Limits for Analysis of Chlorinated Pesticides (EPA 8080/8081).

a 200 mL → 10 mL ∴ **DF = 0.05** (Routine Water)
b Chlorinated Pesticides 10 g → 10 mL = **(1)**
1mL of **(1)** → 10 mL ∴ **DF = 10.0** (Routine Soil)

PCBs 10 g → 10 mL ∴**DF = 1.0** (Routine Soil)

c 1000 mL → 10 mL ∴ **DF = 0.01** (Low Level Water)
d 10 g → 10 mL ∴ **DF = 1.0** (Low Level Soil)
e Dependent on Matrix

ECL

PARAMETER	LO STD CONC (mg/l)	ROUTINE WATER[a] (mg/l)	ROUTINE SOIL[b] (mg/kg)	LO WATER[c] (ug/l)	LO SOIL[d] (mg/kg)	HI WATER&SOIL[e]	DRINKING WATER[f] (ug/l)
Dichlorvos (DDVP)	0.25	0.25	2.5	2.5	0.83	.....	0.50
Mevinphos (Phosdrin)	0.50	0.50	5.0	5.0	1.66	.....	1.0
Sulfotepp (Bladafume)	0.12	0.12	1.2	1.2	0.42	.....	0.25
Thimet (Phorate)	0.25	0.25	2.5	2.5	0.83	.....	0.50
Diazinon	0.25	0.25	2.5	2.5	0.83	.....	0.50
Disyston (Disulfoton)	0.25	0.25	2.5	2.5	0.83	.....	0.50
Parathion Methyl	0.12	0.12	1.2	1.2	0.42	.....	0.25
Ronnel	0.12	0.12	1.2	1.2	0.42	.....	0.25
Malathion	0.25	0.25	2.5	2.5	0.83	.....	0.50
Baytex (Fenthion)	0.12	0.12	1.2	1.2	0.42	.....	0.25
Chlorpyrifos (Lorsban)	0.25	0.25	2.5	2.5	0.83	.....	0.50
Parathion Ethyl	0.12	0.12	1.2	1.2	0.42	.....	0.25
Methidathion m	0.25	0.25	2.5	2.5	0.83	.....	0.50
DEF	0.25	0.25	2.5	2.5	0.83	.....	0.50
Ethion	0.12	0.12	1.2	1.2	0.42	.....	0.25
Trithion	0.25	0.25	2.5	2.5	0.83	.....	0.50
Demeton-O	0.10	0.10	1.0	1.0	0.35	.....	0.21
Ethoprop (Mocap)	0.25	0.25	2.5	2.5	0.83	.....	0.50
Tokuthion	0.25	0.25	2.5	2.5	0.83	.....	0.50
Phosfolan	0.25	0.25	2.5	2.5	0.83	.....	0.50
Fensulfothion	0.25	0.25	2.5	2.5	0.83	.....	0.50
Phosmet	0.25	0.25	2.5	2.5	0.83	.....	0.50
Azinphos Ethyl	0.25	0.25	2.5	2.5	0.83	.....	0.50
Fonofos	0.25	0.25	2.5	2.5	0.83	.....	0.50
Demeton-S	0.40	0.4 0	4.0	4.0	1.3	.....	0.79
Dimethoate	0.25	0.25	2.5	2.5	0.83	.....	0.50
Monocrotophos	0.25	0.25	2.5	2.5	0.83	.....	0.50
Chlorfenvinphos	0.25	0.25	2.5	2.5	0.83	.....	0.50
Leptophos	0.25	0.25	2.5	2.5	0.83	.....	0.50
EPN	0.25	0.25	2.5	2.5	0.83	.....	0.50
Azinphos Methyl (Guthion)	0.25	0.25	2.5	2.5	0.83	.....	0.50
Famphur	0.25	0.25	2.5	2.5	0.83	.....	0.50
Coumaphos	0.25	0.25	2.5	2.5	0.83	.....	0.50

Table 3a. Quantitation Limits for Analysis of Organophosporus Pesticides (EPA 8141).

a 100 mL → 100 mL ∴ **DF = 1** (Routine Water)
b 10 g → 100 mL ∴ **DF = 10** (Routine Soil)
c 1 L → 10 mL ∴ **DF =.01** (Low Level Water)
d 30 g → 100 mL ∴ **DF = 3.33** (Low Level Soil)
e Dependent on Matrix
f 1 L → 2 ml ∴ **DF = .002** (Drinking Water)

ECL-SC

PARAMETER	LO STD CONC (mg/l)	ROUTINE WATER[a] (ug/l)	ROUTINE SOIL[b] (mg/kg)	LO WATER[c] (ug/l)	LO SOIL[d] (mg/kg)	HI WATER&SOIL[e]
Dichlorvos (DDVP)	.....	.....	.....	.....	.....	.....
Naled (Dibrom)	0.50	25	0.50	5.0	0.50	.....
Mevinphos (Phosdrin)	0.50	25	0.50	5.0	0.50	.....
Sulfotepp (Bladafume)	.....	.....	.....	.....	.....	.....
Thimet (Phorate)	0.50	25	0.50	5.0	0.50	.....
Diazinon	0.50	25	0.50	5.0	0.50	.....
Disyston (Disulfoton)	0.50	25	0.50	5.0	0.50	.....
Parathion methyl	0.50	25	0.50	5.0	0.50	.....
Ronnel	.....	.....	.....	.....	.....	.....
Malathion	0.50	25	0.50	5.0	0.50	.....
Baytex (Fenthion)	.....	.....	.....	.....	.....	.....
Chlorpyrifos (Lorsban)	0.50	25	0.50	5.0	0.50	.....
Parathion ethyl	0.50	25	0.50	5.0	0.50	.....
Methidathion	.....	.....	.....	.....	.....	.....
DEF	0.50	25	0.50	5.0	0.50	.....
Ethion	0.50	25	0.50	5.0	0.50	.....
Trithion	0.50	25	0.50	5.0	0.50	.....
Demeton-O	.....	.....	.....	.....	.....	.....
Ethoprop (Mocap)	.....	.....	.....	.....	.....	.....
Tokuthion	.....	.....	.....	.....	.....	.....
Phosfolan	.....	.....	.....	.....	.....	.....
Fensulfothion	.....	.....	.....	.....	.....	.....
Phosmet	.....	.....	.....	.....	.....	.....
Azinphos methyl	.....	.....	.....	.....	.....	.....
Fonofos	.....	.....	.....	.....	.....	.....
Demeton-S	.....	.....	.....	.....	.....	.....
Dimethoate	.....	.....	.....	.....	.....	.....
Monocrotophos	.....	.....	.....	.....	.....	.....
Chlorfenvinphos	.....	.....	.....	.....	.....	.....
Leptophos	.....	.....	.....	.....	.....	.....
EPN	.....	.....	.....	.....	.....	.....
Azinphos methyl (Guthion)	.....	.....	.....	.....	.....	.....
Famphur	.....	.....	.....	.....	.....	.....
Coumaphos	.....	.....	.....	.....	.....	.....
Bolstar (Sulprofos)	.....	.....	.....	.....	.....	.....
Stirophos (Tetrachlorovinphos)	.....	.....	.....	.....	.....	.....

Table 3b. Quantitation Limits for Analysis of Organophosporus Pesticides (EPA 8140).

a 1 L $\rightarrow$ 2 ml $\therefore$ **DF = .002** (Drinking Water)
b 10 g $\rightarrow$ 10 mL $\therefore$ **DF = 1.0** (Routine Soil)
c 1 L $\rightarrow$ 10 mL $\therefore$ **DF =.01** (Low Level Water)
d 10 g $\rightarrow$ 10 mL $\therefore$ **DF = 1.0** (Low Level Soil)
e Dependent on Matrix

ECL

PARAMETER	LO STD CONC (mg/l)	ROUTINE WATER[a] (mg/l)	ROUTINE SOIL[b] (mg/kg)	LO WATER & DRINKING WATER [c] (ug/l)	LO SOIL[d] (mg/kg)	HI WATER & SOIL[e]
2,6 Dinitro Phenol	2.5	0.25	2.5	25	0.82	.....
2,4 Dinitro Phenol	1.2	0.12	1.2	12	0.41	.....
2,5 Dinitro Phenol	1.2	0.12	1.2	12	0.41	.....
3,4 Dinitro Phenol	2.5	0.25	2.5	25	0.82	.....
4,6 Dinitro-o-Cresol (DNOC)	1.2	0.12	1.2	12	0.41	.....
4Nitrophenol	2.5	0.25	2.5	25	0.82	.....
Dinitramine)	1.2	0.12	1.2	12	0.41	.....
Fluchloralin (Basalin)	2.5	0.25	2.5	25	0.82	.....
Trifluralin	2.5	0.25	2.5	25	0.82	.....
Dinoseb	1.2	0.12	1.2	12	0.41	.....
Pendimethalin (Prowl)	2.5	0.25	2.5	25	0.82	.....
Dinocap-1	2.5	0.25	2.5	25	0.82	.....
Dinocap- 2	2.5	0.25	2.5	25	0.82	.....

Table 4. Quantitation Limits for Analysis of Dinitroaromatics by HPLC (ECL 736).

[a] 100 mL → 10 mL ∴ **DF = 0.10** (Routine Water)
[b] 10 g → 10 mL ∴ **DF = 1** (Routine Soil)
[c] 1 L → 10 mL ∴ **DF = 0.01** (Low Level Water & Drinking Water)
[d] 30 g → 10 mL ∴ **DF = 0.333** (Low Level Soil)
[e] Dependent on matrix

ECL

PARAMETERS	LO STD CONC (mg/l)	ROUTINE WATER[a] (mg/l)	ROUTINE SOIL [b] (mg/kg)	LO WATER & DRINKING WATER [c] (ug/l)	LO SOIL [d] (mg/kg)	HI WATER&SOIL[e]
2,4,D Acid	0.25	0.25	2.5	25	0.80	.....
2,4,5-T Acid	0.12	0.12	1.2	12	0.40	.....
2,4-DB Acid	0.25	0.25	2.5	25	0.80	.....
Silvex	0.12	0.12	1.2	12	0.40	.....
Dicamba	0.25	0.25	2.5	25	0.80	.....
Dichloroprop	0.25	0.25	2.5	25	0.80	.....
MCPA	25	25	250	2500	80	.....
Dalapon	0.25	0.25	2.5	25	0.80	.....
Dinoseb	0.25	0.25	2.5	25	0.80	.....
MCPP	25	25	250	2500	80	.....

Table 5. Quantitation Limits for Analysis of Chlorophenoxy Herbicides by GC.

[a] 100 mL → 10 mL = **(1)**
1 ml of **(1)** → 10 ml ∴ **DF = 1** (Routine Water)
[b] 10 g → 10 mL = **(1)**
1 ml of **(1)** → 10ml ∴ **DF = 10** (Routine Soil)
[c] 1 L → 10 ml = **(1)**
1 ml of **(1)** → 10 ml ∴ **DF = 0.1** (Low Level Water & Drinking Water)
[d] 30 g → 10 ml = **(1)**
1 ml of **(1)** →10ml ∴ **DF = 3.33** (Low Level Soil)
[e] Dependent on matrix

ECL

PARAMETER	LO STD CONC (mg/l)	ROUTINE WATER [a] (ug/l)	ROUTINE SOIL [b] (mg/kg)	LO WATER [c] (ug/l)	LO SOIL [d] (mg/kg)	DRINKING WATER [c] (ug/l)
Aldicarb sulfone	0.50	50	0.25	5.0	0.17	5.0
Methomyl (Lannate)	0.50	50	0.25	5.0	0.17	5.0
3-Hydroxycarbofuran	0.50	50	0.25	5.0	0.17	5.0
Dioxacarb	0.50	50	0.25	5.0	0.17	5.0
Aldicarb	0.50	50	0.25	5.0	0.17	5.0
Baygon (Propoxur)	0.50	50	0.25	5.0	0.17	5.0
Carbofuran	0.50	50	0.25	5.0	0.17	5.0
Carbaryl (Sevin)	0.50	50	0.25	5.0	0.17	5.0
Methiocarb (Mesurol)	0.50	50	0.25	5.0	0.17	5.0
Promecarb	0.50	50	0.25	5.0	0.17	5.0

Table 6. Quantitation Limits for Analysis of Carbamates (ECL 734 REV.1).

[a] 100 mL → 100 mL= **(1)**
10 mL of **(1)** → 1mL ∴ **DF = 0.10** (Routine Water)
[b] 20 g → 100 mL = **(2)**
10 mL of **(2)** → 1 mL ∴ **DF = 0.50** (Routine Soil)
[c] 1 L → 100 mL = **(3)**
10 mL of **(3)** → 1 mL ∴ **DF = .01** (Low Level Water & Drinking Water)
[d] 30 g → 100 mL = **(4)**
10 mL of **(4)** → 1mL ∴ **DF = 0.333** (Low Level Soil)

ECL

PARAMETER	LO STD CONC (ug/l)	ROUTINE WATER [a] (ug/l)	ROUTINE SOIL [b] (ug/kg)	LO WATER [c] (ug/l)	DRINKING WATER [d] (ug/l)
EDB	1.0	0.060	10	0.060	0.020
DBCP	0.50	0.030	5.0	0.030	0.010

Table 7. Quantitation Limits for Analysis of EDB and DBCP (EPA 8011).

[a] 35 mL → 2 mL ∴ **DF = 0.06** (Routine Water)
[b] 10 g → 100 mL ∴ **DF = 10** (Routine Soil)
[c] 35 mL → 2 mL ∴ **DF = 0.06** (Low Level Water)
[d] 100 mL → 2 mL ∴ **DF = 0.02** (Drinking Water)

ECL

PARAMETER	LO STD CONC ug/l	WATER [a] ug/l	SOIL [b] ug/kg	DRINKING WATER [c] ug/l
Tetrachlorophenol	100	100	2000	2
Pentachlorophenol	100	100	2000	2

Table 8. Quantitation Limits for Analysis of Chlorophenols (ECL 782)

[a] 100 mL → 50 mL; 5 mL derv. → 50 mL conc. → 10 mL ∴ **DF = 1** (Water)
[b] 10 g → 100 mL; 5 mL derv. → 50 mL conc. → 10 mL ∴ **DF = 20** (Soil)
[c] 1 L → 50 mL; 5 mL derv. → 50 mL conc. → 2 mL ∴ **DF = 0.02** (Drinking Water)

Note: Method ECL 782 is recommended when low level quantitation limits are required for these 2 compounds only.

ECL

PARAMETER	LO STD CONC (ug/l)	ROUTINE WATER[a] (ug/l)	ROUTINE SOIL[b] (mg/kg)	LO WATER[c] (ug/l)	LO SOIL[d] (mg/kg)	HI WATER&SOIL[e]	DRINKING WATER[f] (ug/l)
Naphthalene	30	30	0.3	3.0	0.1	.....	0.3
Acenaphthylene	60	60	0.6	6.0	0.2	.....	0.6
Acenaphthene	40	40	0.4	4.0	0.13	.....	0.4
Fluorene	120	120	1.2	12.0	0.4	.....	1.2
Phenanthrene	10	10	0.1	1.0	0.03	.....	0.1
Anthracene	2	2	0.02	0.2	0.01	.....	0.02
Fluoranthene	10	10	0.1	1.0	0.03	.....	0.1
Pyrene	20	20	0.2	2.0	0.07	.....	0.20
1,2-Benzanthracene	5	5	0.05	0.5	0.016	.....	0.05
Chrysene	5	5	0.05	0.5	0.016	.....	0.05
Benzo(b)fluoranthene	6	6	0.06	0.6	0.02	.....	0.06
Benzo(k)fluoranthene	1	1	0.01	0.1	0.003	.....	0.01
Benzo(a)pyrene	2	2	0.02	0.2	0.01	.....	0.02
Indeno(1,2,3-c,d)pyrene	40	40	0.4	4.0	0.13	.....	0.40
1,2,5,6-Dibenzoanthracene	5	5	0.05	0.5	0.016	.....	0.05
1,12-Benzoperylene	40	40	0.4	4.0	0.13	.....	0.4

Table 9. HPLC Quantitation Limits for Analysis of Polynuclear Aromatic Hydrocarbons (EPA 8310).

[a] 100 mL → 100 mL ∴ **DF = 1.0** (Routine Water)
[b] 10 g → 100 mL ∴ **DF = 10.0** (Routine Soil)
[c] 1 L → 100 mL **DF = 0.1** (Low Level Water)
[d] 30 g → 100 mL **DF = 3.33** (Low Level Soil)
[e] Dependent on Matrix
[f] 1 L → 10 mL **DL = 0.01** (Drinking Water)

ECL

PARAMETER	LO STD CONC (ug/mL)	ROUTINE WATER[a] (ug/mL)	ROUTINE SOIL[b] (mg/kg)	LO WATER[c] (ug/mL)	LO SOIL[d] (mg/kg)	HI WATER&SOIL[e]	DRINKING WATER[f] (ug/l)
Octahydro-1,3,5,7-tetranitro-1,3,5,7-etrazocine(HMX)	0.25	0.25	2.5	0.025	0.83	.....	2.5
Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)	0.25	0.25	2.5	0.025	0.83	.....	2.5
1,3,5-trinitrobenzene(1,3,5-TNB)	0.25	0.25	2.5	0.025	0.83	.....	2.5
Methyl 1-2,4,6-trinitrophenylnitramine (Tetryl)	0.25	0.25	2.5	0.025	0.83	.....	2.5
Nitrobenzene (NB)	0.25	0.25	2.5	0.025	0.83	.....	2.5
2,4,6-Trinitrotoluene (2,4,6-TNT)	0.25	0.25	2.5	0.025	0.83	.....	2.5
4-Amino-2,6-dinitrotolune (4-Am-DNT)	0.25	0.25	2.5	0.025	0.83	.....	2.5
2Amino-4,6-dinitrotolune (4-Am-DNT)	0.25	0.25	2.5	0.025	0.83	.....	2.5
2,4-Dinitrotoluene (2,4-DNT)	0.25	0.25	2.5	0.025	0.83	.....	2.5
2,6-Dinitrotoluene (2,6-DNT)	0.25	0.25	2.5	0.025	0.83	.....	2.5
2-Nitrotoluene(2-NT)	0.25	0.25	2.5	0.025	0.83	.....	2.5
3-Nitrotoluene(3-NT)	0.25	0.25	2.5	0.025	0.83	.....	2.5
4-Nitrotoluene(4-NT)	0.25	0.25	2.5	0.025	0.83	.....	2.5

[a] 100 mL → 100 mL ∴ **DF = 1.0** (Routine Water)
[b] 10 g → 100 mL ∴ **DF = 10.0** (Routine Soil)
[c] 1 L → 100 mL **DF = 0.1** (Low Level Water)
[d] 30 g → 100 mL **DF = 3.33** (Low Level Soil)
[e] Dependent on Matrix
[f] 1 L → 10 mL **DL = 0.01** (Drinking Water)

Table 10. Quantitation Limits for Analysis of Nitroaromatics and Nitramines (EPA 8330)

PARAMETER	LO STD CONC (ug/l)	ROUTINE WATER [a] (mg/l)	ROUTINE SOIL [b] (mg/kg)	LO WATER [c] (ug/l)	LO SOIL [d] (ug/kg)	HI WATER&SOIL [e]
			ECL			
Chloromethane	5.0	0.25	0.62	5.0	5.0	.....
Bromomethane	5.0	0.25	0.62	5.0	5.0	.....
Vinyl chloride	5.0	0.25	0.62	5.0	5.0	.....
Chloroethane	5.0	0.25	0.62	5.0	5.0	.....
Methylene Chloride	5.0	0.25	0.62	5.0	5.0	.....
Trichlorofluoromethane	5.0	0.25	0.62	5.0	5.0	.....
1,1-Dichloroethene	5.0	0.25	0.62	5.0	5.0	.....
1,1-Dichloroethane	5.0	0.25	0.62	5.0	5.0	.....
trans-1,2-Dichloroethene	5.0	0.25	0.62	5.0	5.0	.....
Chloroform	5.0	0.25	0.62	5.0	5.0	.....
1,2-Dichloroethane	5.0	0.25	0.62	5.0	5.0	.....
1,1,1-Trichloroethane	5.0	0.25	0.62	5.0	5.0	.....
Carbon tetrachloride	5.0	0.25	0.62	5.0	5.0	.....
Bromodichloromethane	5.0	0.25	0.62	5.0	5.0	.....
1,2-Dichloropropane	5.0	0.25	0.62	5.0	5.0	.....
trans-1,3-Dichloropropene	5.0	0.25	0.62	5.0	5.0	.....
Trichloroethene	5.0	0.25	0.62	5.0	5.0	.....
Benzene	5.0	0.25	0.62	5.0	5.0	.....
Dibromochloromethane						.....
1,1,2-Trichloroethane	5.0	0.25	0.62	5.0	5.0	.....
cis-1,3-Dichloropropene	5.0	0.25	0.62	5.0	5.0	.....
2-Chloroethylvinyl ether	5.0	0.25	0.62	5.0	5.0	.....
Bromoform	5.0	0.25	0.62	5.0	5.0	.....
1,1,2,2-Tetrachloroethane	5.0	0.25	0.62	5.0	5.0	.....
Tetrachloroethene	5.0	0.25	0.62	5.0	5.0	.....
Toluene	5.0	0.25	0.62	5.0	5.0	.....
Chlorobenzene	5.0	0.25	0.62	5.0	5.0	.....
Ethyl benzene	5.0	0.25	0.62	5.0	5.0	.....
1,3-Dichlorobenzene	.....	.....	.....	.....	.....	.....
1,2-Dichlorobenzene	.....	.....	.....	.....	.....	.....
1,4-Dichlorobenzene	.....	.....	.....	.....	.....	.....
Acetone	20	1.0	2.5	20	20	.....
2-Butanone	20	1.0	2.5	20	20	.....
Carbon disulfide	5.0	0.25	0.62	5.0	5.0	.....
cis 1,3-Dichloropropene	5.0	0.25	0.62	5.0	5.0	.....
2-Hexanone	20	1.0	2.5	20	20	.....
4-Methyl-2-pentanone	20	1.0	2.5	20	20	.....
Styrene	5.0	0.25	0.62	5.0	5.0	.....
Vinyl acetate	20	1.0	2.5	20	20	.....
Xylene (ortho or para)	5.0	0.25	0.62	5.0	5.0	.....
Xylene (meta)	5.0	0.25	0.62	5.0	5.0	.....

[a] 2 mL → 100 mL = (1)
5ml of (1) ∴ Dilution Factor **DF = 50.0** (Routine Water)

[b] 4 g → 10 mL = (1)
0.1ml of (1) to 5ml ∴ Dilution Factor **DF = 125** (Routine Soil)

[c] Direct Purge ∴ **DF = 1** (Low Level Water)

[d] 5 g → 5 mL ∴ **DF = 1** (Low Level Soil) GC/MS purge and trap system is not currently equipped with a heated purging vessel. Method 8260 is being developed to handle low level soil samples.

[e] Dependent on Matrix

Table 11 a. Quantitation Limits for Analysis of GC/MS for Volatile Organics (EPA 8240).

ECL-SC

PARAMETER	LO STD CONC (ug/l)	ROUTINE WATER [a] (mg/l)	ROUTINE SOIL [b] (mg/kg)	LO WATER [c] (ug/l)	LO SOIL [d] (ug/kg)	HI WATER&SOIL [e]
Methylene chloride	5.0	0.25	5.0	5.0	5.0	.....
Freon 113	5.0	0.25	5.0	5.0	5.0	.....
Chloroform	5.0	0.25	5.0	5.0	5.0	.....
1,1,1-Trichloroethane	5.0	0.25	5.0	5.0	5.0	.....
1,2-Dichloroethane	5.0	0.25	5.0	5.0	5.0	.....
Benzene	5.0	0.25	5.0	5.0	5.0	.....
Carbon Tetrachloride	5.0	0.25	5.0	5.0	5.0	.....
Trichloroethylene	5.0	0.25	5.0	5.0	5.0	.....
Toluene	5.0	0.25	5.0	5.0	5.0	.....
Perchlorethylene	5.0	0.25	5.0	5.0	5.0	.....
Chloroethylene	5.0	0.25	5.0	5.0	5.0	.....
Ethylbenzene	5.0	0.25	5.0	5.0	5.0	.....
m & p-Xylene	5.0	0.25	5.0	5.0	5.0	.....
Styrene	5.0	0.25	5.0	5.0	5.0	.....
o-Xylene	5.5	0.25	5.0	5.0	5.0	.....
Cumene	5.0	0.25	5.0	5.0	5.0	.....
0-chlorotoluene	5.0	0.25	5.0	5.0	5.0	.....
n-Propyl Benzene	5.0	0.25	5.0	5.0	5.0	.....
p-Chlorotoluene	5.0	0.25	5.0	5.0	5.0	.....
1,3,5-Trimethylbenzene	5.0	0.25	5.0	5.0	5.0	.....
t-Butylbenzene	5.0	0.25	5.0	5.0	5.0	.....
1,2,4-Trimethylbenzene	5.0	0.25	5.0	5.0	5.0	.....
1,3-Dichlorobenzene	5.0	0.25	5.0	5.0	5.0	.....
sec-Butybenzene	5.0	0.25	5.0	5.0	5.0	.....
1,4-Dichlorobenzene	5.0	0.25	5.0	5.0	5.0	.....
p-Cymene	5.0	0.25	5.0	5.0	5.0	.....
1,2-Dichlorobenzene	5.0	0.25	5.0	5.0	5.0	.....
n-Butylbenzene	5.0	0.25	5.0	5.0	5.0	.....
1,2,4-Ethyl benzene	5.0	0.25	5.0	5.0	5.0	.....
Naphthalene	5.0	0.25	5.0	5.0	5.0	.....
1,2,3-Trichlorobenzene	5.0	0.25	5.0	5.0	5.0	.....
Acetone	40	2.0	400	40	40	.....
Methyl isobutyl Ketone	40	2.0	400	40	40	.....
Methyl Ethyl Ketone	40	2.0	400	40	40	.....
1,1-Dichloroethyene	5.0	0.25	5.0	5.0	5.0	.....
1,2-Dichloroethylene (T)	5.0	0.25	5.0	5.0	5.0	.....
1,1,Dichloroethane	5.0	0.25	5.0	5.0	5.0	.....
1,2-Dichloroethylene ©	5.0	0.25	5.0	5.0	5.0	.....
1,1-Dichloropropene	5.0	0.25	5.0	5.0	5.0	.....
1,2-Dichloropropene	5.0	0.25	5.0	5.0	5.0	.....
Bromodichloromethane	5.0	0.25	5.0	5.0	5.0	.....
1,3-Dichloropropene ©	5.0	0.25	5.0	5.0	5.0	.....
1,3-Dichloropropene (T)	5.0	0.25	5.0	5.0	5.0	.....

[a] 2 mL → 100 mL = (1)
5ml of (1) ∴ Dilution Factor **DF = 50.0** (Routine Water)

[b] 10 g → 10 mL = (1)
50 uL of (1) to 50 mL = (2)
25 mL of (2) ∴ Dilution Factor **DF = 1000** (Routine Soil)

[c] Direct Purge ∴ **DF = 1** (Low Level Water)

[d] 5 g → 5 mL ∴ **DF = 1** (Low Level Soil)

[e] Dependent on Matrix

Table 11 b. Quantitation Limits for Analysis of GC/MS for Volatile Organics (EPA 8260) Cont..

ECL-SC

PARAMETER	LO STD CONC (ug/l)	ROUTINE WATER [a] (mg/l)	ROUTINE SOIL [b] (mg/kg)	LO WATER [c] (ug/l)	LO SOIL [d] (ug/kg)	HI WATER&SOIL[e]
1,1-2-Trichloroethane	5.0	0.25	5.0	5.0	5.0	.....
1,3-Dichloropropane	5.0	0.25	5.0	5.0	5.0	.....
Dibromochloromethane	5.0	0.25	5.0	5.0	5.0	.....
Ethylenedibromide	5.0	0.25	5.0	5.0	5.0	.....
1,1,1,2-Tetrachloroethane	5.0	0.25	5.0	5.0	5.0	.....
Bromoform	5.0	0.25	5.0	5.0	5.0	.....
1,1,2,2-Tetrachloroethane	5.0	0.25	5.0	5.0	5.0	.....
1,2,3-Trichloropropane	5.0	0.25	5.0	5.0	5.0	.....
Hexachlorobutadiene	5.0	0.25	5.0	5.0	5.0	.....
Vinyl Chloride	5.0	0.25	5.0	5.0	5.0	.....

[a] 2 mL → 100 mL = (1)
5ml of (1) ∴ Dilution Factor **DF = 50.0** (Routine Water)

b 10 g → 10 mL = (1)
50 uL of (1) to 50 mL = (2)
25 mL of (2) ∴ Dilution Factor **DF = 1000** (Routine Soil)

[c] Direct Purge ∴ **DF = 1** (Low Level Water)

[d] 5 g → 5 mL ∴ **DF = 1** (Low Level Soil)

[e] Dependent on Matrix

Table 11 b. Quantitation Limits for Analysis of GC/MS for Volatile Organics (EPA 8260).

TABLE 12 ESTIMATED QUANTITATION LIMITS FOR GC/MS ANALYSIS OF SEMIVOLATILE ORGANICS (EPA 8270) at ECL and ECL-SC[1,2]

Parameters	CAS No.	Standard[3] (µg/mL)	Low Water[4a] (µg/L)	Routine Water[4b] (mg/L)	Low Soil[5a] (mg/Kg)	(mg/Kg)
						5
acenaphthene	83-32-9	5	5	0.25	0.67	5
acenaphthylene	208-96-8	5	5	0.25	0.67	10
aniline	62-53-3	10	10	0.5	1.33	5
anthracene	120-12-7	5	5	0.25	0.67	5
benz[a]anthracene	56-55-3	5	5	0.25	0.67	
						5
benzo(a)pyrene	50-32-8	5	5	0.25	0.67	5
benzo(b)fluoranthene	205-99-2	5	5	0.25	0.67	5
benzo(g,h,i)perylene	191-24-2	5	5	0.25	0.67	5
benzo(k)fluoranthene	207-08-9	5	5	0.25	0.67	10
benzyl alcohol	100-51-6	10	10	0.5	1.33	
						5
bis(2-chloroethoxy)methane	111-91-1	5	5	0.25	0.67	5
bis(2-chloroethyl)ether	111-44-4	5	5	0.25	0.67	5
bis(2-chloroisopropyl)ether	108-60-1	5	5	0.25	0.67	5
bis(2-ethylhexyl)phthalate	117-81-7	5	5	0.25	0.67	5
4-bromophenyl phenyl ether	l01-55-3	5	5	0.25	0.67	
						5
buty benzyl phthalate	85-68-7	5	5	0.25	0.67	5
carbazole	86-74-8	5	5	0.25	0.67	10
4-chloro-3-methylphenol	59-50-7	10	10	0.5	1.33	10
4-chloroaniline	106-47-8	10	10	0.5	1.33	5
2-chloronaphthalene	91-58-7	5	5	0.25	0.67	
						10
2-chlorophenol	95-57-8	10	10	0.5	1.33	5
4-chlorophenyl phenyl ether	7005-72-3	5	5	0.25	0.67	5
chrysene	218-01-9	5	5	0.25	0.67	10
dibenz[a,h]anthracene	53-70-3	10	10	0.5	1.33	10
dibenzofuran	132-64-9	10	10	0.5	1.33	
						5
di-n-butyl phthalate	84-74-2	5	5	0.25	0.67	5
1,2-dichlorobenzene	95-50-1	5	5	0.25	0.67	5
1,3-dichlorobenzene	541-73-1	5	5	0.25	0.67	5
1,4-dichlorobenzene	106-46-7	5	5	0.25	0.67	20
3,3'-dichlorobenzidine	91-94-1	20	20	1	2.67	
						10
2,4-dichlorophenol	120-83-2	10	10	0.5	1.33	5
diethyl phthalate	84-66-2	5	5	0.25	0.67	5
dimethyl phthalate	131-11-3	5	5	0.25	0.67	10
2,4-dimethylphenol	105-67-9	10	10	0.5	1.33	50
4,6-dinitro-2-methylphenol	534-52-1	50	50	2.5	6.67	
						50
2,4-dinitrophenol	51-28-5	50	50	2.5	6.67	5
2,4-dinitrotoluene	121-14-2	5	5	0.25	0.67	5
2,6-dinitrotoluene	606-20-2	5	5	0.25	0.67	5
di-n-octyl phthalate	117-84-0	5	5	0.25	0.67	5
fluoranthene	206-44-0	5	5	0.25	0.67	
						5
fluorene	86-73-7	5	5	0.25	0.67	
	Lowest				**Routine Soil[5b]**	

TABLE 12 ESTIMATED QUANTITATION LIMITS FOR GC/MS ANALYSIS OF SEMIVOLATILE ORGANICS (EPA 8270) at ECL and ECL-SC (con't)

Parameter	CAS No.	Lowest Standard[3] (µg/mL)	Low Water[4a] (µg/L)	Routine Water[4a] (mg/L)	Low Soil[5a] (mg/Kg)	Routine Soil[5b] (mg/Kg)
hexachlorobenzene	118-74-1	5	5	0.25	0.67	5
hexachlorobutadiene	87-68-3	5	5	0.25	0.67	5
hexachlorocyclopentadiene	770-47-4	5	5	0.25	0.67	5
hexachloroethane	67-72-1	5	5	0.25	0.67	5
indeno(1,2,3-c,d)pyrene	193-39-5	5	5	0.25	0.67	5
isophorone	78-59-1	5	5	0.25	0.67	5
2-methylnaphthalene	91-57-6	5	5	0.25	0.67	5
2-methylphenol	95-48-7	10	10	0.5	1.33	10
(4 & 3)-methylphenol	106-44-5	10	10	0.5	1.33	10
naphthalene	91-20-3	5	5	0.25	0.67	5
nitobenzene	98-95-3	5	5	0.25	0.67	5
2-nitroaniline	88-74-4	20	20	1	2.67	20
3-nitroaniline	99-09-2	20	20	1	2.67	20
4-nitroaniline	l00-01-6	20	20	1	2.67	20
2-nitrophenol	88-75-5	10	10	0.5	1.33	10
4-nitrophenol	100-02-7	50	50	2.5	6.67	50
n-nitrosodimethylamine[6]	62-75-9	10	10	0.5	1.33	10
n-nitrosodiphenylamine	86-30-6	5	5	0.25	0.67	5
n-nitrosodipropylamine	621-64-7	5	5	0.25	0.67	5
pentachlorophenol	87-86-5	50	50	2.5	6.67	50
phenanthrene	85-01-8	5	5	0.25	0.67	5
phenol	108-95-2	10	10	0.5	1.33	10
pyrene	129-00-0	5	5	0.25	0.67	5
pyridine[6]	110-86-1	5	5	0.25	0.67	5
1,2,4-trichlorobenzene	120-82-1	5	5	0.25	0.67	5
2,4,5-trichlorophenol	95-95-4	20	20	1	2.67	20
2,4,6-trichlorophenol	88-06-2	10	10	0.5	1.33	10

Footnotes:

[1]..... Table 12 lists semivolatile organic analytes that are routinely analyzed at both ECL laboratories with GC/MS Method 8270. The estimated quantitation limits (EQLs) serve as a guide for the proper selection of a determinative method. The decision should be based on the method's ability to meet quantiation limits required by the analytical objective (i.e., regulatory limits, project specific quantitation limits, etc.). Generally, mass spectrometry is more specific for qualitative identification, but it is often less sensitive than determinative methods that are based on compound specific detectors (i.e., ECD for chlorinated semivolatile organics, HPLC/UV for polynuclear aromatic hydrocarbons, etc.). Therefore when the quantitatiion limit requirements exceed those achievable with Method 8270, it may be necessary to employ a more sensitive determinative method and confirmed with second chromatographic column or detector.

[2]ECL's quantitation limit (QL) is defined as the lowest standard concentration used in the method's calibration table multiplied by the sample's dilution factor and a matrix factor.

[3]The Estimated Quantitation Limits (EQLs) are highly matrix -dependent. The EQLs listed above are provided for guidance and may not always be achievable. For low level soil and water samples, EQLs are estimated assuming minimal chemical and physical interferences.

[4]EQLs for water samples are estimated using extraction procedure of "EPA Method 3510 -Separatory Funnel Extraction"

a) For low water samples, EQLs assume a one liter volume of sample is extracted and concentrated to a final volume of one milliliter.
b) For routine water samples, 200 milliliters of a water sample is extracted and concentrated to a final volume of ten milliliters.

[5]EQLs for soil samples are estimated using extraction procedure of "Method 3540 - Soxhlet Extraction." EQLs are estimated based on wet weight therefore EQLs will be higher if based on dried weight basis.

a) For low soil samples, EQLs assume a thirty grams sample soil sample is extracted to a final volume of four milliliters.
b) For routine soil samples, ten grams of soil is extracted and concentrated to a final volume of ten milliliters

[6]Pyridine and n-nitrosodimethylamine are not routine target analytes. These are determined upon special request and should be approved by ECL prior to sample submittal. For semivolatile organic analytes not listed above, please contact ECL's GCMS Laboratories.

	ECL		**ECL-SC**		
PARAMETER	WATER [a] (ug/l)	SOIL [a] (ug/kg)	WATER[b] (mg/l)	LO SOIL[b] (mg/kg)	HI SOIL[b] (mg/kg)
Ag-Silver 10 50 0 0 0	10	50	1.0	5.0	50
_____	30	100	1.0	5.0	50
Ba-Barium _____	2	10	1.0	5.0	50
Be-Beryllium _____	2	5	0.10	1.0	5.0
Cd-Cadmium _____	3	10	0.10	1.0	5.0
Co-Cobalt _____	10	50	1.0	5.0	50
Cr-Chromium _____	15	80	1.0	5.0	50
Cu-Copper _____	20	100	1.0	5.0	50
Hg-Mercury _____	.....	.....	.....	.....	.....
Mo-Molybdenum _____	10	100	1.0	5.0	50
Ni-Nickel _____	10	50	1.0	5.0	50
Pb-Lead _____	30	100	1.0	5.0	50
Sb-Antimony _____	30	150	1.0	5.0	50
Se-Selenium _____	40	150	0.10	1.0	5.0
Tl-Thallium _____	50	200	1.0	5.0	50
V-Vanadium	10	50	1.0	5.0	50
_____					
Zn-Zinc _____	20	100	1.0	5.0	50

a Thermo Jarrel Ash (Simultaneous ICP)
[b] Jobin-Yvon JY-50 (Simultaneous ICP)

Table 13. Method Detection Limits for Analysis of Metals (EPA 6010).

	ECL			ECL-SC	
PARAMETER	LO STD CONC (ug/l)	LO WATER [a] (ug/l)	LO SOIL [b] (mg/kg)	WATER (ug/l)	LO SOIL[c] (mg/kg)
Ag-Silver	0.50	0.50	0.025	0.50	1.0
As-Arsenic	5.0	5.0	0.25	5.0	…….....
Ba-Barium	2.0	2.0	0.10	2.0	…….....
Be-Beryllium	0.20	0.20	0.010	0.20	0.50
Cd-Cadmium	0.10	0.10	0.0050	0.10	0.50
Co-Cobalt	1.0	1.0	0.050	1.0	5.0
Cr-Chromium	1.0	1.0	0.0010	1.0	5.0
Cu-Copper	1.0	1.0	0.050	1.0	5.0
Mo-Molybdenum	1.0	1.0	0.050	1.0	5.0
Ni-Nickel	1.0	1.0	0.050	1.0	5.0
Pb-Lead	5.0	5.0	0.25	1.0	5.0
Sb-Antimony	3.0	3.0	0.15	3.0	…….....
Se-Selenium	3.0	3.0	0.15	3.0	…….....
Tl-Thallium	1.0	1.0	0.050	1.0	5.0
V-Vanadium	4.0	4.0	0.20	4.0	5.0
Zn-Zinc	…….....	…….....	…….....	0.50	1.0

[a] 100 mL → 100 mL ∴ Dilution Factor **(DF)** = **1**
[b] 2 g → 100 mL ∴ Dilution Factor **(DF)** = **50**
[c] Flame Atomic Absorption

Table 14. Quantitation Limits for Analysis of Metals (EPA 7000 series Furnace methods).

	ECL			ECL-SC	
PARAMETER	LO STD CONC. (ug/l)	LO WATER [a] (ug/l)	LO SOIL [b] (mg/kg)	WATER (mg/l)	SOIL (mg/kg)
Hg-Mercury	0.50	0.50	0.05	0.01	0.5

[a] 100 mL → 100 mL ∴ Dilution Factor **(DF)** = **1** (EPA 7470)
[b] 1 g → 100 mL ∴ Dilution Factor **(DF) = 100** (EPA 7471)

Table 15. Quantitation Limits for Analysis of Mercury (Cold Vapor).

	ECL			ECL-SC	
PARAMETER	LO STD CONC. (mg/l)	LO WATER [a] (mg/l)	LO SOIL [b] (mg/kg)	WATER (ug/l)	SOIL (ug/kg)
Fluoride	0.50	0.50	5.0	…….....	…….....
Chloride	0.50	0.50	5.0	…….....	…….....
Sulfate	1.5	1.5	15	…….....	…….....
Nitrate	1.5	1.5	15	…….....	…….....
Phosphate	0.20	0.20	2.0	…….....	…….....

[a] 100 mL → 100 mL ∴ Dilution Factor **(DF)** = **1**
[b] 10 g → 100 mL ∴ Dilution Factor **(DF) = 10**

Table 16. Quantitation Limits for Analysis of Anions (ECL 960).

		ECL			ECL SC	
PARAMETER	LO STD CONC (mg/l)	WATER [a] (mg/l)	LO STD CONC (mg/l)	SOIL [b] (mg/kg)	WATER (ug/Kg)	SOIL (ug/Kg)
Cyanide	0.20	0.10	0.50	2.5	…….....	…….....

[a] 500 mL → 250 mL ∴ **Dilution Factor (DF) = 0.5**
[b] 50 g → 250 mL ∴ Dilution Factor **(DF) = 5**

Table 17. Quantitation Limits for Analysis of Cyanide (EPA 9010).

To: Liaisons

From: Nabil Yacoub
Department Lead Liaison

Date: August 7, 1996

Subject: **Common Laboratory Contaminants**

ISSUE: Reviewers of data packages have, on various occasions, encountered different levels of volatile and semivolatile compounds reported by some RP labs as laboratory contaminants. In some instances, the compounds were not detected in the blanks. The issue is the identity of the compounds, volatile and semivolatile, that ECL would consider as common lab contaminants, and whether field sampling equipment are contributing to the problem.

ECL RESPONSE: (based on discussions in the ECL's SOP Committee)

The USEPA-CLP [1] has identified the following compounds as common lab contaminants detected in the analysis for volatile and semivolatile organics:

CLP - Volatiles	CLP - Semivolatiles
Methylene Chloride	Common Phthalate Contaminants
Acetone	
2-Butanone	

ECL concurs, and further identifies the following Phthalates as possible lab contaminants:

bis-2 Ethylhexyl Phthalate	n-Butyl Phthalate
Diethyl Phthalate	n-Octyl Phthalate
Benzyl Phthalate	

ECL and other laboratories have also identified Chloroform as a contaminant when analyzing for volatile organic compounds.

When analyzing samples, such as drinking water, for low levels of metals, Si and Al are often

detected as common contaminants absorbed from the air.

To determine the existence and magnitude of contamination resulting from laboratory (or field) activities, the CLP requires laboratories to analyze a number of lab and field **BLANKS.** CLP then sets criteria for the evaluation of data, and specifies actions to be taken when certain levels of contaminants are detected. The Venn diagram (Figure 1. Below) [2] show some blanks which may be used in the sampling/analytical process. An equipment blank, for example, which is intended to measure the cleanliness of the sampling equipment, could potentially be contaminated in the field, during transportation to the lab, or in the laboratory itself. A method blank, on the other hand, could only be contaminated during sample preparation and analysis in the lab.

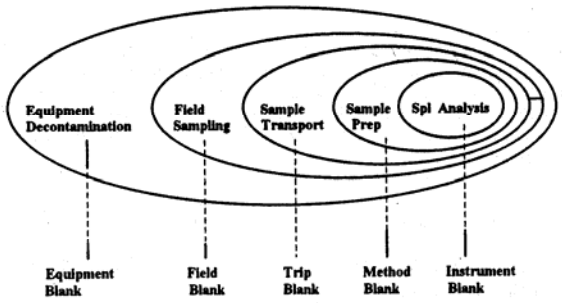


Figure 1 - Blank Samples and Artificially Introduced Contaminants

CLP requires laboratories to apply a set of specific criteria when contaminants are detected in the blanks. Consultants should consider these criteria before releasing a data package. Reviewers of data packages should also be aware of the criteria when reviewing the packages. Information included in the attached excerpts from the CLP National Functional Guidelines for Organic Data Review [1] detail the objectives, criteria, evaluation, and action to be taken in the analysis of volatile and semivolatile organics by CLP laboratories (Attachments).

In general, action regarding unsuitable blank results depends on the circumstances and origin of the blank. Positive volatile and semivolatile sample results should be reported unless the concentration of the compound in the sample is less than or equal to 10 times (10 x) the amount in any blank for the common laboratory contaminants, or 5 times (5x) the amount for other target compounds. In instances where more than one blank is associated with a given sample, qualification should be based on a comparison with the associated blank having the highest concentration of a contaminant. The results must NOT be corrected by subtracting any blank value. However, all sample data should be qualified when possible sample contamination might have occurred.

In the inorganic analysis for metals, the 5x criteria apply. If sample results are greater than the instrument Detection Limit (IDL) but less than 5 times the amount found in any blank, results should be qualified as (U). The value is either the sample quantitation limit or the sample detection limit [3].

Sources of contamination may vary laboratory solvents and water, and powdered gloves to lab and field equipment. Analysis of lab and field blanks may help identify these sources of contamination.

Professional judgment is essential particularly when reviewing problematic data packages. Blank and sample data must be carefully evaluated to determine whether or not there is an inherent variability in the data, or if the problem is an isolated occurrence not affecting other data.

If you encounter a problematic data package, you may solicit the expert help of ECL staff to provide the needed evaluation. This service has been frequently used by project managers from various activities especially from the Office of Military Facilities (OF). P.A. and Site codes, and a completed Work Form would be required.

Contact Myrto Petreas at (510) 540-3624 or mpetreas@dtsc.ca.gov.

References:

(1) USEPA-CLP Program, National Functional Guidelines for Organic Data Review, February 1994.

(2) USEPA-Region IX, RCRA Corrective Action, Data Review Guidance Manual, July, 1995.

(3) USEPA-CLP Program, National Functional Guidelines for Inorganic Data Review, February 1994.

Attachments

cc: Bart Simmons, Ph.D.
Bob Stephens, Ph.D.

V. Blanks

A. Review Items: Form I VOA (Form I LCV), Form IV VOA (Form IV LCV), chromatograms, and quantitation reports.

B. Objective:

The purpose of laboratory (or field) blank analysis is to determine the existence and magnitude of contamination resulting from laboratory (or field) activities. The criteria for evaluation of blanks apply to any blank associated with the samples (e.g., method blanks, instrument blanks, trip blanks, and equipment blanks). If problems with any blank exist, all associated data must be carefully evaluated to determine whether or not there is an inherent variability in the area, or if the problem is an isolated occurrence not affecting other data.

C. Criteria:

1. No contaminants should be found in the blanks.

2. A method blank analysis must be performed after the calibration standards and once for every 12-hour time period beginning with the injection of BFB.

3. The method blank must be analyzed on each GC/MS system used to analyze sample for each type of analysis, i.e., unheated purge (water and medium level soil) and heated purge (low level soil).

4. A storage blank must be prepared upon receipt of the first samples from an SDG, and stored with samples until analysis. The storage blank must be analyzed once per SDG.

5. An instrument blank must be analyzed after any sample that has saturated ions from a given compound to check that blank is free of interference and the system is not contaminated.

D. Evaluation:

1. Review the results of all associated blanks on the forms and raw data (chromatograms and quantitation reports) to evaluate the presence of target and non-target compounds in the blanks.

2. Verify that a method analysis has been reported per matrix, per concentration level, for each 12-hour time period on each GC/MS system used to analyze volatile samples. The reviewer can use the Method Blank Summary (Form IV VOA/form IV LCV) to identify the samples associated with each method blank.

3. Verify that a storage blank has been analyzed and included with each SDG and that the storage blanks are free of contamination.

4. Verify that the instrument blank analysis has been performed following any sample analysis where a target analyte(s) is/are reported at high concentration(s).

Action:

If the appropriate blanks were not analyzed with the frequency described in Criteria 2,3, and 4, and 5 then the data reviewer should use professional judgment to determine if the associated sample data should be qualified. The reviewer may need to obtain additional information from the laboratory. The situation should be noted for TPO action.

Action regarding unsuitable blank results depends on the circumstances and origin of the blank. Positive sample results should be reported unless the concentration of the compound in the sample is less than or equal to 10 times (10x) the amount in any blank for the common volatile laboratory contaminants (methylene chloride, acetone, and 2-butanone), or 5 times (5x) the amount for other volatile target compounds. In instances where more than one blank is associated with a given sample, qualification should be based upon a comparison with the associated blank having the highest concentration of a contaminant. The results must <u>not</u> be corrected by subtracting any blank value.

Specific actions are as follows:

1. If a volatile compound is found in a blank but not found in the sample, no action is taken. If the contaminants found are volatile target compounds (or interfering non-target compounds) at significant concentrations above the CRQL, then this should be noted for TPO action.

2. Any volatile compound detected in the sample (other than the common volatile laboratory contaminants), that was also detected in any associated blank, is qualified if the sample concentration is less than five times (5x) the blank concentration. The quantitation limit may also be elevated. Typically, the sample CRQL is elevated to the concentration found in the sample. The reviewer should use professional judgment to determine if further elevation of the CRQL is required. For the common volatile laboratory contaminant the results are qualified by elevating the quantitation limit to the concentration found in the sample when the sample concentration is less than 10 times (10x) the blank concentration.

   The reviewer should note that blanks may not involve the same weights, volumes, or dilution factors as the associated samples. These factors must be taken into consideration when applying the "5x" and "10x" criteria, such that a comparison of the total amount of contamination is actually made.

   Additionally, there may be instances where little or no contamination was present in the associated blanks, but qualification of the sample is deemed necessary. If the reviewer determines that the contamination is from a source other than the sample, he/she should qualify the data. Contamination introduced through dilution water is one example. Although it is not always possible to determine, instances of this occurring can be detected when contaminants are found in the diluted sample result, but are absent in the undiluted sample result. Since both results are not routinely reported, it may be impossible to verify this source of contamination. In this case, the "5x" or "10x" rules may not apply;
   The target compound should be reported as not detected, and an explanation of the data qualification should be provided in the data review narrative.

3. If gross contamination exists (i.e., saturated by GC/MS), all affected compounds in the associated samples should be qualified as unusable(R) due to interference. This should be noted for TPO action if the contamination is suspected of having an effect on the sample results.

4. If inordinate number of other target compounds are found at low levels in the blank(s), it may be indicative of a problem and should be noted for TPO action.

5. The same consideration given to the target compounds should also be given to Tentatively Identified Compounds (TICs), which are found in both the sample and associated blank(s). (See VOA Section XII for TIC guidance.)

6. If contaminants are found in the storage blanks, the following action is recommended.

   a. The associated method blank data should be reviewed to determine if the contaminant(s) was also present in the method blank. If the analyte was present at a comparable level in the method blank, then the source of the contamination may be in the analytical system and the action recommended for the method blank would apply.

      If the analyte was not present in the method blank, then the source of contamination may be in the storage and all associated samples should be considered for possible cross-contamination.

   b. If the storage blank contains a volatile TCL compound(s) at a concentration greater than the CRQL, then all positive results for that compound(s) should be qualified with “J”. If the concentration level in the blank is significantly high, then positive sample results may require rejection and be qualified with “R”. Non-detected volatile target compounds should not require qualification unless the contamination is so high that it interferes with the analysis of the non-detect compounds.

7. If an instrument blank was not analyzed following a sample analysis which contained an analyte(s) at high concentration(s), sample analysis results after the high concentration sample must be evaluated for carryover. Professional judgment should be used to determine if instrument cross-contamination has affected any positive compound identification(s). If instrument cross-contamination is suspected of having an effect on the sample results.

The following are examples of applying the blank qualification guidelines. Certain circumstances may warrant deviations from these guidelines.

Example 1: Sample result is greater than the Contract Required Quantization Limit (CRQL), but is less than the 5x or 10x multiple of the blank result.

	Rule	
	10x	5x
Blank Result	7	7
CRQL	5	5
Sample Result	60	30

Final Sample Result	60U	30U

In the example for the "10x" rule, sample results less than 70 (or 10x7) would be qualified as not detected. In the case of the "5x" rule, sample results less than 35 (or 5x7) would be qualified as not detected.

Example 2: Sample result is less than the CRQL, and is also less than the 5x or 10x Multiple of the blank result.

	Rule	
	10x	5x
Blank Result	6	6
CRQL	5	5
Sample Result	4J	4J
Final Sample Result	5U	5U

Not that data are not reported as 4U, as this would be reported as a detection limit below the CRQL.

Example 3: Sample result is greater than the 5x or 10x multiple of the blank result.

	Rule	
	10x	5x
Blank Result	10	10
CRQL	5	5
Sample Result	120	60
Final Sample Result	120	120

For both the "10x" and "5x" rules, sample results exceeded the adjusted blank results of 100 (or 10x10) and 50 (or 5x10), respectively, and therefore are not qualified.

VI . System Monitoring Compounds

A. Review Items: Form II VOA (From II LCV), quantization reports, and chromatograms.

B. Objective:

Laboratory performance on individual samples is established by means of spiking activities. All sample are spiked with system monitoring compounds, SMC, (formerly referred to as surrogates) just prior to sample purging. The evaluation of the results of these system monitoring compounds is not necessarily straightforward. The sample itself may produce effects due to such factors as interferences and high concentrations of analytes. Since the effects of the sample matrix are frequently outside the control of the laboratory and may present relatively unique problems, the evaluation and review of data based on specific sample results is frequently subjective and demands analytical experience and professional judgment. Accordingly, this section consists primarily of guidelines, in some cases with several optional approaches suggested.

C. Criteria:

1. Three system monitoring compounds (1,2-dichloroethane-d4, bromofluorobenzene, and toluene-d8) are added to all samples and blanks to measure their recovery in environmental samples in sample and blank matrices.

   (For data generated through the Low Concentration Water Method: A single system monitoring compound, bromofluorobenzene, is added to all samples and blanks to measure the recovery in sample and blank matrices)

2. Recoveries for system monitoring compounds in volatile samples and blanks must be within the limits specified in the Method.

D. Evaluation:

1. Check raw data (e.g., chromatograms and quantization reports) to verify the recoveries on the System Monitoring Compound Recovery Form - Form II VOA (Form II LCV). Check for any calculation or transcription errors.

2. Check that the system monitoring compound recoveries were calculated correctly. The equation can be found in the Method.

3. The following should be determined from the System Monitoring Compound Recovery form(s):

V.Blanks

A. Review Items: Form I SV-1 and SV-2 (Form 1 LCSV-1 and LCSV -2), Form IV SV (Form IV LCSV), chromatograms, and quantization reports.

B. Objective:

The purpose of laboratory (or field) blank analyses is to determine the existence and magnitude of contamination problems resulting from laboratory (or field) activities. The criteria for evaluation of blanks apply to any blank associated with the samples (e.g., method blanks, instrument blanks, trip blanks, and equipment blanks). If problems with any blank exist, all associated data must be carefully evaluated to determine whether or not there is an inherent variability in the data, or if the problem is an isolated occurrence not affecting other data.

C. Criteria:

1. No contaminants should be found in the blanks.

2. The method blank must be analyzed on each GC/MS system used to analyze that specific group or set of samples.

D. Evaluation:

1. Review the results of all associated blank, Form I SV-1 and SV-2, and raw data (chromatograms and quantization reports) to evaluate the presence of target and non-target compounds in the blanks.

2. Verify that a method blank analysis has been reported per matrix, per concentration level, for each extraction batch and for each GC/MS system used to analyze semivolatile samples. The reviewer can use the method blank summary (Form IV SV) to assist in identifying samples associated with each method blank.

E. Action:

If the appropriate blanks were not analyzed with the frequency described above, then the data reviewer should use professional judgment to determine if the associated sample data should be qualified. The reviewer may to obtain additional information from the laboratory. The situation should be noted for TPO action.

Action in the case of unsuitable blank results depends on the circumstances and origin of the blank. Positive sample results should be reported unless the concentration of the compound in the sample is less than or equal to 10 times (10x) the amount in any blank for the common phthalate contaminants, or 5 times the amount for other compounds. In instances where more than one blank is associated with a given sample, qualification should be based upon a comparison with the associated blank having the highest concentration of a contaminant. The results must not be corrected by subtracting any blank value.

Specific actions are as follows:

6. If a volatile compound is found in a blank but not found in the sample, no action is taken. If the contaminants found are volatile target compounds (or interfering non-target compounds) at significant concentrations above the CRQL, then this should be noted for TPO action.

7. Any semivolatile compound detected in the sample (other than the common volatile laboratory contaminants), that was also detected in any associated blank, is qualified if the sample concentration is less than five times (5x) the blank concentration. The quantization limit may also be elevated. Typically, the sample CRQL is elevated to the concentration found in the sample. The reviewer should use professional judgment to determine if further elevation of the CRQL is required. For phthalate contaminants, the results are qualified "U" by elevating the quantization limit to the sample concentration when the sample result is less than 10 x the blank concentration.

   The reviewer should note that blanks may not involve the same weights, volumes, or dilution factors as the associated samples. These factors must be taken into consideration when applying the "5x" and "10x" criteria, such that a comparison of the total amount of contamination is actually made.

   Additionally, there may be instances where little or no contamination was present in the associated blanks, but qualification of the sample was deemed necessary. Contamination introduced through dilution water is one example. Although it is not always possible to determine, instances of this occurring can be detected when contaminants are found in the diluted sample result, but are absent in the undiluted sample result. Since both results are not routinely reported, it may be impossible to verify this source of contamination. However, if the reviewer determines that the concentration is from a source other than the sample, he/she should qualify the data. In this case, the "5x" or "10x" rules may not apply; the

sample value should be reported as a non-detect. An explanation of the rationale used for this determination should be provided in the narrative accompanying the Regional Data Assessment Summary.

3. If gross contamination exists (i.e., saturated by GC/MS), all affected compounds in the associated samples should be qualified as unusable(R) due to interference.
This should be noted for TPO action if the contamination is suspected of having an effect on the sample result.

4. If inordinate number of other target compounds are found at low levels in the blank(s), it may be indicative of a problem and should be noted for TPO action.

5. The same consideration given to the target compounds should also be given to Tentatively Identified Compounds (TICs), which are found in both the sample and associated blank(s). (See SV Section XIII for TIC guidance.)

The following are examples of applying the blank qualification guidelines. Certain circumstances may warrant deviations from these guidelines.

Example 1: Sample result is greater than the Contract Required Quantization Limit (CRQL), but is less than the 5x or 10x multiple of the blank result.

	Rule	
	10x	5x
Blank Result	12	12
CRQL	10	10
Sample Result	50	40
Qualified Sample Result	50U	40U

In the example for the “10x” rule, sample results less than 120 (or 10x12) would be qualified as non- detected. In the case of the “5x” rule, sample results less than 60 (or 5x12) would be qualified as non-detects.

Example 2: Sample result is less than the CRQL, and is also less than the 5x or 10x Multiple of the blank result.

	Rule	
	10x	5x
Blank Result	12	12
CRQL	10	10
Sample Result	8J	8J
Qualified Sample Result	10U	10U

Not that data are not reported as 8U, as this would be reported as a detection limit below the CRQL.

Example 3: Sample result is greater than the 5x or 10x multiple of the blank result.

	Rule	
	10x	5x
Blank Result	15	15
CRQL	10	10
Sample Result	160	80
Qualified Sample Result	160	160

For both the “10x” and “5x” rules, sample results exceeded the adjusted blank results of 150 (or 10x15) and 75 (or 5x15), respectively, and therefore are not qualified.

VI. Surrogate Spikes

F. Review Items: Form II SV-1 and SV-2 (Form II LCSV), chromatograms, and quantitation reports.

B. Laboratory performance on individual samples is established by means of spiking activities. All sample are spiked with surrogate compounds, prior to sample preparation. The evaluation of the results of these surrogate spikes is not necessarily straightforward. The sample itself may produce effects due to such factors as interferences and high concentrations of analytes. Since the effects of the sample matrix are frequently outside

the control of the laboratory and may present relatively unique problems, the evaluation and review of data based on specific sample results is frequently subjective and demands analytical experience and professional judgment. Accordingly, this section consists primarily of guidelines, in some cases with several optional approaches suggested.

C. Criteria:

1. Surrogate spikes, 4 acid compounds (3 required and 1 advisory) and 4 base/neutral compounds (3 required and 1 advisory) are added to all samples and blanks to measure their recovery in sample and blank matrices.
(*For data generated through the Low Concentration Method: Surrogate spikes, 3 acid compounds and 3 base/neutral compounds, are added to all samples and blanks to measure their recovery in sample and blank matrices.)*

2. Surrogate spike recoveries for semivolatile samples and blanks must be within the limits specified on in the SOW and on Form II SV-1 and SV-2.

(*For data generated through the Low Concentration Method: Surrogate spike recoveries for semivolatile samples and blanks must be within the limits specified in the method and on Form II LCSV)*

D. Evaluation:

5. Check raw data (e.g., chromatograms and quantitation reports) to verify the surrogate spike recoveries on the Surrogate Recovery Form II SV-1 and SV-2 (Form II LCSV). Check for any transcription or calculation errors.

6. Check that the surrogate spike recoveries were calculated correctly. The equation can be found in the method.

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Section 3.2.3 Surface water was revised by Christopher Guerre, Senior Hazardous Substances Engineering Geologist.